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## ENGINEERING DATA TRANSMITTAL

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# **Data Quality Objectives Process Summary, Sampling and Analysis Plan, and Quality Assurance Project Plan in Support of Group 2b Waste Disposition**

Prepared for the U.S. Department of Energy  
Assistant Secretary for Environmental Management  
*Project Hanford Management Contractor for the*  
U.S. Department of Energy under Contract DE-AC06-96RL13200

**Fluor Hanford**

P.O. Box 1000  
Richland, Washington

# Data Quality Objectives Process Summary, Sampling and Analysis Plan, and Quality Assurance Project Plan in Support of Group 2b Waste Disposition

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# **Data Quality Objectives Process Summary, Sampling and Analysis Plan, and Quality Assurance Project Plan in Support of Group 2b Waste Disposition**

October 2001

Prepared by

**Environmental Quality Management**  
for  
**The Plutonium Finishing Plant**

**Executive Summary**

The Group 2b items, which are part of the inventory of plutonium alloy residues currently stored at the Plutonium Finishing Plant (PFP), are considered to have the potential for designation as Dangerous Waste. Group 2b consist of 38 items categorized as Turnings in Oil, Skulls, Chips, and Turnings, Not-specified scrap, Pu/Zr Alloy, Sludge, Sweeps, and Plastic Mounts. Fluor Hanford has evaluated the items contained in this inventory to determine the most appropriate method for characterizing the Group 2b items to support waste designation. Laboratory personnel at the PFP performed a limited evaluation of three items to determine their stability. This analysis determined that the items were stable on exposure to air, water, and heat (up to 95°C). Conducting these analyses resulted in a significant dose to the PFP personnel.

Analysis performed on alloys similar to the Group 2b items in the early 1960s indicated that there is potential for the Group 2b items to contain Resource Conservation and Recovery Act of 1976/Dangerous Waste Toxicity Characteristic (TC) metals at concentrations that approximate the regulatory threshold. Although project staff originally planned to conduct sampling and analysis of items to determine the concentrations of TC metals, because of concerns over personnel exposure, the decision was made to stipulate for the presence of TC metals for all Group 2b items. This designation will not affect the ultimate disposal of the materials as debris at the Waste Isolation Pilot Plant (WIPP).

The inventory description listed some of the items as Turnings in Oil. This description led the project staff to initially consider sampling the oils to evaluate them for the presence of volatile organics and polychlorinated biphenyls (PCBs). The stability evaluation, referenced above, revealed that oil is present only as a hardened, residual coating on the container and some of the turnings. The condition of the oil indicates that there are no volatile organic compounds present. A preliminary solubility test of the oil from the Turnings in Oil suggests that the substance has a Lard Oil base, which is consistent with materials practices when machining plutonium. Although the results from the solubility test cannot be considered conclusive, they provide a strong basis due to the apparent non-polar nature of oils. The presence of Lard Oil argues against the presence of PCBs because (1) Lard Oil possesses the same heat resistant

characteristics as PCBs, (2) PCBs would likely cause pitting and staining of the alloy, and (3) scrap material was recycled into the process – use of PCBs would have contaminated the subsequent melt. If sufficient oil (1 g) can be removed from the Turnings in Oil to conduct analysis, the oil will be evaluated for PCBs. If there is not sufficient oil to perform the analysis, the assumption will be made that the oil is Lard Oil and there are no PCBs in the Group 2b items.

*Sludge will be analyzed to evaluate this item for the characteristic of corrosivity.*

All items are considered to be non-reactive and not ignitable, based on management history of the Group 2b items, the results of the stability evaluation, and the composition and form of the alloy items.

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**List of Acronyms**

ACES	Access Control Entry System
Al	Aluminum
ALARA	As Low As Reasonably Achievable
BNWL	Battelle Northwest Laboratories
COPC	Constituent of Potential Concern
DOE	United States Department of Energy
DQO	Data Quality Objectives
EPA	Environmental Protection Agency
EQL	Estimated Quantitation Limit
GC	Gas Chromatography
HASQARD	Hanford Analytical Services Quality Assurance Requirements Documents
LC	Lard Can
LCS	Laboratory Control Sample
MDL	Method Detection Limit
PCB	Polychlorinated Biphenyl
PFP	Plutonium Finishing Plant
PHMC	Project Hanford Management Contract
PPSL	Plutonium Process Support Laboratories
PQL	Practical Quantitation Limit
PRTR	Plutonium Recycle Test Reactor
Pu	Plutonium
QA	Quality Assurance
QC	Quality Control
RCRA	Resource Conservation and Recovery Act of 1976
RL	Richland Operations Office
SAP	Sampling and Analysis Plan
SS	Stainless Steel
SVOC	Semi-Volatile Organic Compound
TC	Toxicity Characteristic
TRU	Transuranic
TSCA	Toxic Substances Control Act
VOC	Volatile Organic Compound
WIPP	Waste Isolation Pilot Plant

## 1 PLUTONIUM ALLOYS - GROUP 2B DATA QUALITY OBJECTIVES

### 1.1 INTRODUCTION

In the 1950s, the Atomic Energy Commission initiated work to evaluate the use of plutonium (Pu) as a fuel for power reactors. This work continued until the mid-1970s, and included work that was performed in the 308 Building at the Hanford Site. Materials from the fuel fabrication processes that contained significant amounts of Pu were stored at the Plutonium Finishing Plant (PFP) pending recovery of the Pu. Residual materials from these activities remain in storage at the PFP, including scrap materials from the fabrication of Pu alloy fuels.

The PFP inventory includes 126 items classified as Pu alloy scrap and residue. The items consist primarily of Pu metal alloyed with aluminum (Al) for experimental reactor fuel and scrap materials from the fabrication of these alloys. The alloy materials (Al and zirconium), after fabrication with Pu, were used to produce reactor fuel. The residual alloy items include both special nuclear material, in the form of the plates, rods, and billets that were fabricated into fuel, and byproduct materials from the fabrication process.

Thirty-eight of the items in the Pu alloy group are byproduct material that potentially contain hazardous/dangerous waste constituents, based on the material form and/or the possibility of regulated constituents in the waste matrix. These items are collectively labeled Group 2b and are the subject of this Data Quality Objectives (DQO) process and Sampling and Analysis Plan (SAP). The Group 2b items listed on the PFP inventory include the following residues from various stages in the fabrication process:

- Turnings in Oil (2 items)
- Skulls, Chips, and Turnings (15 items)
- Not Specified Pu/Al Scrap (16 items)
- Pu/Zr Alloy (2 items)
- Sludge (1 item)
- Sweeps (1 item)
- Plastic Mounts (1 item)

Table 1 presents a listing of the various Group 2b items, along with the descriptions of the items as found in the PFP inventory (Borisch et. al. 2001). Descriptions provided in Table 1 are reproduced as they appear in the PFP inventory; the precise meaning of some of the acronyms used is not clear based on available information. This DQO document addresses the evaluation of hazardous/dangerous waste and Toxic Substances Control Act (TSCA) issues associated with these Group 2b items.

Table 1. Group 2b Item Descriptions (2 pages)

Subgroup	Item	CAT Description	COEI Description	Description-History Files
Casting/skulls	CE-3-80-9-1	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu in Cryolite Rpkgd (entry only for -80-9)
Casting/skulls	CE-3-80-9-2	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu in Cryolite Rpkgd (entry only for -80-9)
Casting/skulls	CE-3-80-9-3	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu in Cryolite Rpkgd (entry only for -80-9)
Casting/skulls	CE-3-80-9-4	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu in Cryolite Rpkgd (entry only for -80-9)
Casting/skulls	CE-3-80-6-1	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu in Cryolite Rpkgd (entry only for -80-6)
Casting/skulls	CE-3-80-6-3	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu in Cryolite Rpkgd (entry only for -80-6)
Casting/skulls	CE-3-80-6-2	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu in Cryolite Rpkgd (entry only for -80-6)
Casting/skulls	CE-6-605-4	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Pu AL Casting skull
Casting/skulls	CE-6-605-3	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Pu AL Casting Skull, X ray available
Chips/Turnings	60-438-2	BNW Pu-Al Scrap FG other	Pu alloy (impure)	Saw chips, turnings, Al turnings
Chips/Turnings	60-438-1	BNW Pu-Al Scrap FG other	Pu alloy (impure)	Saw chips, turnings, Al turnings
Chips/Turnings	CE-3-76-1-5	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu Saw Chips - (entry for -76-1)
Chips/Turnings	CE-3-76-1-2	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu Saw Chips - (entry for -76-1)
Chips/Turnings	CE-3-76-1-1	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu Saw Chips - (entry for -76-1)
Chips/Turnings	CE-3-90-3	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	PuAl Chips, misc, scraps, + sweeps, X ray available + O56
Dried Sludge	CE-3-80-8	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	AL Pu in Dried Sludge NaOH
Not Specified Pu/Al Scrap	CE-10-1-8-4	BNW Misc Scrap FG 04	Pu alloy (impure)	
Not Specified Pu/Al Scrap	CE-10-1-8-5	BNW Misc Scrap FG 04	Pu alloy (impure)	
Not Specified Pu/Al Scrap	CE-10-1-8-1	BNW Misc Scrap FG 04	Pu alloy (impure)	
Not Specified Pu/Al Scrap	CE-10-1-8-2	BNW Misc Scrap FG 04	Pu alloy (impure)	
Not Specified Pu/Al Scrap	CE-6-602-24	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	20.5% PuAl
Not Specified Pu/Al Scrap	AL-33-12-11	Pu rec Misc Scrap FG	Pu nonconforming alloy	
Not Specified Pu/Al Scrap	AL-33-12-10-4-1	Pu rec Misc Scrap FG	Pu nonconforming alloy	PuO2 from Al rods (-4 was split into -4-1 & -4-2)

**Table 1. Group 2b Item Descriptions (2 pages)**

Subgroup	Item	CAT Description	COEI Description	Description-History Files
Not Specified Pu/Al Scrap	AL-33-12-10-4-2	Pu rec Misc Scrap FG	Pu nonconforming alloy	PuO <sub>2</sub> from Al rods (-4 was split into -4-1 & -4-2)
Not Specified Pu/Al Scrap	AL-33-12-10-3	Pu rec Misc Scrap FG	Pu nonconforming alloy	X ray available
Not Specified Pu/Al Scrap	33-75-06-581	Pu rec Misc Scrap FG	Nonconforming alloy	
Not Specified Pu/Al Scrap	LC-84-06-02	Waste Drums for Burial	Pu alloy (impure)	
Not Specified Pu/Al Scrap	AL-33-12-05	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	
Not Specified Pu/Al Scrap	AL-33-12-07	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	
Not Specified Pu/Al Scrap	AL-33-12-06	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	
Not Specified Pu/Al Scrap	AL-33-12-03	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	X ray available
Not Specified Pu/Al Scrap	AL-33-12-08	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	
Plastic Mounts/Carbid e	63-155-1	BNW Misc Scrap WG	Pu alloy (impure)	PuC/U, PuO <sub>2</sub> Waste, Plastic mounts and Xbles
Pu-Zirconium	61-453-5	Pu Zirc scrap 04	Pu alloy (impure)	Al Zr 2.5% Scrap
Pu-Zirconium	61-453-7	Pu Zirc Scrap 04	Pu alloy (impure)	Al ZR 2.5% Scrap, x ray available
Sweeps	CE-3-80-7	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	Al Pu Hood Sweeps, X ray available
Turnings in Oil	60-518	BNW Pu-Al Scrap FG 04	Pu alloy (impure)	LC in 55 gal, T Pu 7.4% Turnings SS (physical inspection show oil when opened to Rpkg 1980) ~ Metal Turnings in oil
Turnings in Oil	60-436	BNW Pu-Al Scrap FG other	Pu alloy (impure)	LC in 55 gal, T Pu 7.4% Turnings SS (physical inspection show oil when opened to Rpkg 1980) ~ Metal Turnings in oil

## 1.2 DQO PROCESS

### 1.2.1 Problem statement

#### 1.2.1.1 Process History

Experimental fuel fabrication activities at the Hanford Site took place primarily in the Plutonium Fabrication Pilot Plant in the 308 Building, located in the 300 Area just north of Richland. The fabrication of Pu alloy fuel elements supported studies in the Plutonium Recycle Test Reactor (PRTR) in the 300 Area, and also provided fuels for other United States Department of Energy (DOE) sites. Battelle Northwest Laboratories (BNWL) fabricated Pu fuel materials with Al and other alloys; between 1960 and 1963 alone, over 6500 fuel components were processed in the 308 Building (HW-SA-2904). Based on the PFP inventory records, the majority of alloy

residues in storage at PFP were shipped to PFP from BNWL activities conducted in the 308 Building (Borisch 2001). The sources of materials are recorded on inventory lists and labels on individual canned items.

Metallurgical and mechanical processes employed for the fabrication of Pu/Al alloy fuels included, for example, furnace melting and casting, extrusion, forging, pressing, drawing, and machining. Graphite molds and crucibles were used because they required no lubrication (e.g., Wick 1967). Pu/Al scrap material from the process normally was recycled to recover the Pu for reuse. Descriptions of the alloying process in reference texts (e.g., Wick 1967, Coffinberry 1961) indicate the processes used would have been mechanical in nature; i.e., chemical processes were not used in the fabrication procedure. Although there was no chemical processing of the metals, some references discuss the use of organic solvents for degreasing metals used in the fabrication of alloys in other processes or locations. Even though these references do not specifically describe the fabrication process that resulted in the alloy materials stored at PFP, they may provide an indication of what might have been the customary procedures when working with alloy and fuels fabrication.

- Freshley (1961a) refers to the use of trichloroethylene as a degreaser for final cleaning of sheathing tubes. This step in the process, however, is downstream from the process stage that would have resulted in the alloy materials that are in storage (see also, HW-69200 PT 2).
- Freshley (1961b) refers to wiping the "cores clean with trichloroethylene saturated gauze." These cores could have been produced in the same time period as feed material for the Group 2b items, but, again, would have been a downstream process.
- Sharp (1960) refers to the cleaning of cans and lids with trichloroethylene prior to tinning, for some foils and tins that were developed at Hanford for the Savannah River Site. These items, however, are not part of the PFP inventory.
- Bailey (1959) notes that "all coextrusion billet parts were degreased with trichloroethylene." The processing of the items referenced in this document predated the existence of the 308 Building.
- Wauchope (1961) speaks of cleaning finished slugs of alloy with carbon tetrachloride. The reference discusses alloy preparation for a reactor in Canada. This operation provided a model for early alloy work at Hanford (see e.g., Freshley 1957); however, the inventory of Pu alloys is from later alloy work.

In summary, the cited references indicate that degreasing solvents had a history of use in the fabrication of alloys and fuel materials. These materials, however, generally predate the activities at the 308 Building. None of the references reviewed provide a detailed description of processes used specifically for the experimental alloys fabrication or a direct connection between degreasing activities and the materials that are in storage at PFP. Although the original design of the 308 Building incorporated a vapor degreaser process (e.g., Merker 1963a), one of the managers of early operations at the 308 Building recalled that the process was never used. The same individual indicated in an interview that the facility did not use solvents or lubricants in any of its processes (Merker 2001). This recollection is not inconsistent with the documents cited above, which reflect activities that took place before construction of the Building 308 facility or activities that did not contribute feed to this material stream. Use of lubricants on the Pu/Al alloy was found to cause pitting and staining of the metal surface, which was not a desirable side effect

(Wick 1967). Elimination of the lubricants would have eliminated much of the need for degreasing. When a lubricant was used to machine plutonium, Lard Oil was generally used (Kingsley 1962). Bloomster (1960) notes that casting techniques were developed for aluminum-plutonium alloys to save time and reduce scrap generation; graphite molds were used because they require no lubrication. A co-extrusion technique was also developed to reduce the amount of machining (Bloomster 1960).

The following process information is excerpted from a description of Pu/Al fabrication from the Plutonium Handbook (Wick 1967). O.J. Wick, the editor of the Plutonium Handbook and author of the section on Pu/Al fabrication, was employed at Pacific Northwest Laboratory, and it is reasonable to expect that he was familiar with the fabrication process. There is no reason to believe that the processes conducted at the 308 Building would have differed significantly from what is described below.

*Aluminum-plutonium alloys are melted in graphite or clay-graphite crucibles at 800 to 950°C in air and cast at 725 to 800°C into graphite molds. Graphite was preferred because there was no need to use lubricants as a release agent. Casting skulls and metal scrap were re-melted under cryolite and recovered for use without chemical reprocessing.*

[A note is made under a description of the extrusion process that the use of a lubricant on the billet is unnecessary. In fact, the use of a lubricant "caused black spots in the extrusion which could not be easily removed and seemed to initiate galling" (Wick 1967). This notation provides a strong basis to believe that the process would not have used lubricants, due to concerns over product quality.]

*Breakdown of cast ingots was usually performed at temperatures of 500 to 600°C and roll bonding of aluminum-plutonium alloys to aluminum cladding was done at similar temperatures.*

*The fabrication process involved the melting of 1500 grams of reactor grade aluminum at 900°C; 210 grams of metallic plutonium in the form of cast rods were added to the melt to produce a 14 wt. % alloy. After an hour at 900°C, the melt temperature was reduced to 700°C and the alloy was cast into three 1.25-inch diameter x 8-inch long rods. The cast rods were cropped and samples taken at each end for plutonium analysis. Scrap was recycled to the melt. After the plutonium analysis of each rod was obtained, they were cut into cylindrical forging billets – each containing a nominal 8.16 grams of plutonium and weighing 55 grams.*

*Cast billets were forged into shape in dies heated to 400°C using 125 tons of pressure. Acceptable billets were cleaned through the following steps: wire brush, etching with a solution of 10% HNO<sub>3</sub> - 2% HF, water rinse, ultrasonic cleaning in a detergent solution, flushing with water, drying with trichloroethylene, and a smear test for alpha contamination. Clean billets were assembled with plates, welded, and heated in a furnace at 520°C for 10 hours.*

*Unrolled assemblies were heated at 590°C for a minimum of 2-1/2 hours, then reduced with a rolling mill through multiple passes. Plates were reheated between passes and excess end material was sheared away before further processing. Plates were heated at 500°C for 30 minutes and at 600°C for 30 minutes and then examined for blisters and alpha contamination. Elements were machined to ensure precise fit within the fuel assemblies.*

This information is consistent with the recollection of L. Merker, and suggests that solvents were not used at the facility for degreasing purposes. The net result of this review, while inconclusive as to whether degreasing solvents were used in the fabrication of the alloy materials under investigation, supports an assumption that they were not routinely employed in the processes that contributed to the alloy materials under consideration in this DQO process.

#### 1.2.1.2 Constituents of Potential Concern

Based on the description of the alloy fabrication process, as provided above, constituents of potential concern (COPCs) to be evaluated in the alloys are discussed below. The following sections discuss the waste form and analytical limitations, consider the various materials that would have been used in the fabrication process, the material form of the items and the history of the items that are being evaluated, and provide a basis for further evaluation.

##### 1.2.1.2.1 Alloy Metal Constituents

Fuels and weapons grade Pu both were carefully refined to assure that trace metal concentrations were low, so as to not affect reactor or weapon performance. Analyses of alloy materials similar to the Group 2b items, performed to evaluate the qualitative effects of irradiation on fuel materials, showed the presence of chromium, silver, and possibly lead at less than trace concentrations (Freshley 1961c). These analyses were qualitative assessments only, and were not intended to demonstrate compliance with regulatory standards. Although the values used to designate "trace" may translate to levels that approximate the regulatory threshold concentrations (Table 2), the records indicate that detected concentrations were less than trace quantities. These results are consistent with the purity concentrations specified for Pu.

Specifications for Pu buttons included upper limit concentrations for "impurity elements". These criteria included, for example, upper limits for cadmium at 10 ppm, chromium at 100 ppm, and lead at 100 ppm (Dienes 1985). As illustrated in Table 2, buttons containing the maximum allowable levels of chromium and lead would have concentrations of those constituents that match their respective Toxicity Characteristic (TC) limits<sup>1</sup> (100 ppm); the maximum allowed value for cadmium would result in a concentration below the TC limit (20 ppm). Al was selected as an alloying metal in part because it could be obtained in a high-purity form. Addition of the alloying metal would most likely have reduced the concentrations of TC contaminants that might have been present in the Pu.

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<sup>1</sup> TC criteria are provided as the action level based on analysis for total metals; the TC limit is multiplied by 20 for comparison to the total metals results.

Table 2. TC Metals Impurities in Pu Alloys and Buttons (1 page)

Element	Qualitative Spectrographic Data <sup>1</sup>					Extrapolated Metals Concentrations (ppm) <sup>2</sup>	Upper Limit Impurity Levels (ppm) <sup>3</sup>	TC limit *20 (ppm) <sup>4</sup>												
	Alloy Item																			
	GEH-14-272 GEH-14-273	GEH-14-274 GEH-14-275 GEH-14-276 GEH-14-277	GEH-14-278	GEH-14-279	GEH-14-287 GEH-14-288															
Arsenic	----	----	----	----	----	----	NS	100												
Barium	----	----	----	----	----	----	NS	2000												
Cadmium	----	----	----	----	----	----	10	20												
Chromium	----	T	<T	<T	----	<100	100	100												
Lead	*	----	----	----	*	<100	100	100												
Mercury	----	----	----	----	----	----	NS	4												
Silver	----	----	----	----	T	<100	NS	100												
<table><tr><td><u>Symbol</u></td><td><u>Meaning</u></td><td><u>Symbol</u></td><td><u>Meaning</u></td></tr><tr><td>T</td><td>Trace – <u>Less than</u> 0.01%</td><td>----</td><td>Data were not reported for this analyte.</td></tr><tr><td>*</td><td>Interference</td><td>NS</td><td>Not Specified</td></tr></table>									<u>Symbol</u>	<u>Meaning</u>	<u>Symbol</u>	<u>Meaning</u>	T	Trace – <u>Less than</u> 0.01%	----	Data were not reported for this analyte.	*	Interference	NS	Not Specified
<u>Symbol</u>	<u>Meaning</u>	<u>Symbol</u>	<u>Meaning</u>																	
T	Trace – <u>Less than</u> 0.01%	----	Data were not reported for this analyte.																	
*	Interference	NS	Not Specified																	

<sup>1</sup> Data from Freshley (1961c) (The Reactivity of High-Exposure Plutonium). These analyses are for data similar to, but not from, Group 2b alloys.

<sup>2</sup> Based on "Trace" meaning < .01 %. Lead was assumed present at trace amounts.

<sup>3</sup> Data from Dienes (1985) (Specifications for Plutonium Shipped to the Rocky Flats Plant).

<sup>4</sup> Action Level for Total Metals Analysis. EPA allows comparison of total metals results to TC limits by multiplying the TC limit by 20 (SW-846 Method 1311).

#### 1.2.1.2.2 Stability/Reactivity/Ignitability/Corrosivity

Pu metal reacts with water and organic matter (including plastics) through the process of radiolysis, generating hydrogen gas. If contained under pressure, hydrogen gas can pose hazards. The available information for the Pu/Al items suggests that solvents, degreasers, and other organic compounds were not used in the fabrication processes that resulted in the Group 2b items. This conclusion indicates that there should be no basis for concern over the presence of volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), herbicides, or pesticides in those waste items that are clearly composed of alloy material. Available knowledge indicates that generally Lard Oil, or possibly "Fab Oil", a blend of Lard Oil and carbon tetrachloride, was generally used in the machining of Pu. Lard Oil was used because it has a high tolerance for heat, making it desirable for use as a machining lubricant or coolant. Carbon tetrachloride was used because it also has a high tolerance for heat. In addition, because carbon tetrachloride is volatile at ordinary temperatures, its use as a coolant "eliminates the necessity of processing large volumes of liquid waste for the recovery of plutonium" (Wick 1967).

Any VOC or low molecular weight SVOC constituents that may have been present in the containers would have dissipated or been radiolyzed over the 30+ years that the materials have been in storage. Observation of a sample from the Turnings in Oil items confirms that residual oil present in the containers has dried to a hardened, shellac-like coating on the metal items and the container wall (Photos 1 and 2, Appendix 1). This condition suggests that there is no basis for a concern over the presence of TC volatile or semi-volatile organic compounds. In addition, an evaluation of three Group 2b items in the Plutonium Process Support Laboratory (PPSL) found that these items are very stable when exposed to ambient air, temperatures up to 95°C, and when immersed in water (Cooper 2001). This evaluation indicates that these items do not possess the characteristics for ignitability or reactivity.

Pure Pu metal fines can spontaneously ignite in the presence of oxygen at temperatures above room temperature, whereas large metal pieces will not burn unless heated to red-hot temperatures. Because the alloy items contain only low concentrations of Pu metal, the items are not considered to present any ignitability concerns. The management history of the items confirms that there have been no reactive metals problems associated with the alloys; this aspect of the material properties is confirmed by the limited testing performed at PFP. Skulls, which are the thin alloy residue left behind in the crucibles, were thought to present a potential concern, based largely on the thickness of the material. Preliminary evaluations of skull items indicate that they exist as a solid, metallic form and do not present a reactivity hazard (Photo 3, Appendix 1) (Cooper 2001). All items should be opened within a glovebox, however, and observed for any reaction on exposure to air to confirm their status.

The sludge is described in the inventory as dried sodium hydroxide. This description raised the possibility that the sludge could designate for the characteristic of corrosivity.

### **1.2.1.2.3 VOCs, SVOCs, and PCBs**

A determination must be made through process knowledge or analysis whether VOCs, SVOCs, and/or PCBs are present in any oils in order to support the waste designation profile. Oil that was used to store turnings could cause the item to designate due to the presence of VOCs or SVOCs, including PCBs (under TSCA). A review of the uses for the TC SVOC constituents indicates that they are not likely to have been used in the fabrication of alloys (Appendix 2).

As noted above, the lubricant of choice for working with Pu metal at the Hanford Site was Lard Oil. Lard Oil was used in large part because of its heat-resistant qualities. Although PCBs are also heat-resistant, Lard Oil was typically used with Pu because it is a fatty material that does not contain chlorides, which pit metals. As noted elsewhere in this document, lubricants were generally avoided that created staining or pitting of the alloy material. Because the PCBs are generally added to oils for their heat resistance properties, there would have been no reason to add them to the Lard Oil. In addition, the scrap alloy material was routinely recycled to the melt to recover plutonium. It is unlikely that the additions of PCBs to the process would have been beneficial to the goal of a high-purity alloy. This conclusion was supported in an interview with a former plutonium metals worker who stated that there is no reason to think or believe that PCBs would have been added to the Lard Oil (L. Oates interview with J. Teal, October 3, 2001). In addition, a research study conducted in 1961 looked at over 16 different lubricants to use in tapping unalloyed plutonium metal (Rector and Weihermiller 1961). This study concluded that a mixture of perchloroethylene and Lard Oil was the optimal mixture for this purpose. Nowhere in this study is there a discussion of the use of PCBs. Based on the considerations listed, there is little reason to believe that PCBs would be present in the oils associated with the alloys.

### **1.2.1.3 Problem Statement**

The process descriptions for these items indicate that they were generated from production of Special Nuclear Material. Group 2b materials appear to fall within the definition of Byproduct Material under the Atomic Energy Act. Therefore, although the radionuclides themselves are not subject to regulation as hazardous/dangerous wastes, these Byproduct Materials may contain additional hazardous/dangerous or PCB constituents that would cause them to designate as mixed waste or TSCA mixed waste. The Group 2b items must be evaluated to determine whether hazardous/dangerous waste constituents are present in specific waste categories.

### **1.2.1.4 Summary of Problem Statement**

The Group 2b items require additional evaluation and/or characterization to support designation in order to ensure appropriate onsite storage and management, and to develop a refined inventory of the waste constituents prior to shipment to the Waste Isolation Pilot Plant (WIPP) for disposal.

## **1.2.2 Step 2 – Principal Study Questions**

Principal study questions help to phrase the problem statement in a manner that establishes a basis for making decisions.

1. Do the Group 2b items contain oils with (TC) VOCs?
2. Do the Group 2b items contain regulated concentrations of the Resource Conservation and Recovery Act of 1976 (RCRA)/Dangerous waste TC metals?
3. Do any of the Group 2b items present a basis for concern over reactivity?
4. Do any of the Group 2b items contain PCBs at concentrations that would be regulated under TSCA?
5. Is the pH of the sludge in the range that will cause it to exhibit the characteristic of corrosivity?

These questions must be addressed for each of the subgroups within Group 2b, as appropriate. The evaluation will be iterative for each subgroup of items, as appropriate for the subgroup; that is, each question must be answered in turn for each subgroup, as appropriate for that subgroup. A positive response to any of the relevant study questions (i.e., detection of regulated constituents above action levels) will result in management of the subgroup as transuranic (TRU)-mixed waste and/or TRU-TSCA waste. The evaluation must consider all relevant study questions, even after a positive response, in order to develop a complete waste profile for each subgroup.

The following section lists the alternative actions that result from answering "yes" or "no" to the above study questions. Actions are numbered to match the study questions.

#### **1.2.2.1 Alternative Actions**

- 1A) Oil that is present in the subgroup contains TC volatile organic constituents above regulated concentrations. Determine and implement appropriate management/treatment procedures for items in this subgroup as TRU-mixed waste.
- 1B) Oil does not contain TC volatile organic constituents at actionable concentrations. Manage as TRU-only waste.
- 2A) Items contain TC metals at concentrations that exceed action limits. Manage as TRU-mixed waste.
- 2B) TC metals are not present at concentrations that exceed action limits. Manage as TRU-only waste.
- 3A) A visual evaluation or operational history indicates that an item is potentially reactive. Designate and manage as TRU-mixed waste.
- 3B) Evaluation does not indicate a reason for concern over reactivity. Determine and implement appropriate waste management/treatment procedures for items in this subgroup, based on remaining criteria.
- 4A) PCBs are present at concentrations that exceed action levels. Designate and manage the items in subgroup as TRU-mixed (TSCA) waste.
- 4B) PCBs are not present above regulated concentrations. Manage as TRU-only waste.

5A) Sludge has a pH less than or equal to 2 or greater than or equal to 12.5. Manage as a corrosive, TRU- mixed waste.

5B) Sludge has a pH between 2 and 12.5. Manage as a TRU-only waste.

Based on the preceding questions and alternative actions, the following decision statements apply to the Group 2b.

#### **1.2.2.2 Decision Statements**

1. Evaluate whether any oil packaged with the Turnings in Oil, Sludge, or Sweeps potentially contains TC VOC constituents.
2. Determine whether TC metals are present in concentrations that exceed action limits for all items in Subgroup 2b.
3. Determine, based on observation and management history, whether Group 2b items within specific subgroups present a basis for designation as reactive.
4. Determine whether representative samples for subgroups within Group 2b contain PCB constituents at concentrations that exceed TSCA action limits.
5. Determine whether the Sludge requires designation for the characteristic of corrosivity.

#### **1.2.3 Step 3 – Required Inputs**

The purpose of the input section is to identify information, both available and to be gathered, that will be used in the evaluation of the Group 2b items and to list the analytical methods and associated action limits for use in evaluation of the subject material.

##### **1.2.3.1 As Low As Reasonably Achievable (ALARA) Concerns**

All of the items evaluated through this DQO process are byproducts from the fabrication of Pu-based fuel materials. These items each contain weight percent concentrations of Pu and have the potential to cause a high dose exposure to personnel who handle these materials. As noted in Section 1.2.12.2, the PFP laboratory conducted a limited stability evaluation of a subset of the Group 2b items in Spring of 2001 (Feb to May). Table 3 illustrates the dose rates that were recorded for these items, as reported by Ewalt (2001), along with the exposure limits established by DOE and Fluor Hanford. Contact readings listed in Table 3 represent the dose exposure at the surface of the container.

The technicians who performed the majority of this work each received an average 350-mrem whole body dose and 1800-mrem exposure to their extremities. This dose can be compared to the limits established by DOE and Fluor Hanford administrative criteria for worker exposure, as presented in Table 3. Fluor's administrative limits correspond to those levels recorded at 30 cm from the source item.

The Sludge and Sweeps items in the inventory contain plutonium content similar to that found in the items that have been evaluated at PFP. Based on this comparison, it is reasonable to expect that workers handling these items will receive a dose similar to that experienced during analysis of those items listed in Table 3.

**Table 3. Worker Exposure and Dose Limits (1 page)**

Exposure to Workers Evaluating Pu Alloys			Dose Limits	
Item	Contact (Surface of the Container)	30 cm	DOE Limit	Fluor Limit (Administrative, HNF-5173)
Chips (CE-3-76-1-1)	2.5 rem/hr	95 mrem/hr		
Skulls, Chips, Turnings (CE-3-890-6-1)	3.5 rem/hr	160 mrem/hr		
Turnings in Oil	507 mrem/hr	7 mrem/hr		
Total Dose			Annual Limits	
Whole Body		350 mrem	5 rem/year	500 mrem/year
Eyes			15 rem/year	4500 mrem/year
Extremities		1,800 mrem	50 rem/year	15,000 mrem (1.5 rem)/year

Although trace quantities of some regulated metals may be present in some of the items (Table 2), concentrations are expected to be below regulatory levels. Because of the ALARA concerns associated with sampling and analysis of the Pu/Al items, sampling is not considered to be appropriate for a determination of the presence of metals in these items. Because the limited data that are available for alloy materials indicate the presence of selected TC metals at concentrations that potentially approach the regulated limits, sampling and analysis would be necessary to demonstrate conclusively that the metals are not present. Because these items are considered to be debris, for purposes of disposal at the WIPP, addition of the metals waste code will not affect their long-term management. Although a designation for TC metals would normally require analysis for the underlying hazardous constituents, because these items will be going to WIPP, this analysis will not be required for disposal (WIPP Waste Acceptance Criteria). Mercury, although not reasonably anticipated in the majority of the items, could be present in the sweeps due to the potential for broken thermometers in a glovebox.

#### 1.2.3.2 Evaluation of the Analytical Reporting Limits

Observations of the oil in one of Turnings in Oil containers indicate that the oil has formed a hardened, shellac-like coating on the interior and contents of this container (Photos 1 and 2, Appendix 1). This waste form would be highly unlikely to designate for VOC constituents; any volatiles that might have been present clearly have dissipated. If oil is present in a low-viscosity form (i.e., it is in a liquid state) in any item, the item will be stipulated as containing TC VOC constituents. Because the solidified oil that has been observed would have to be diluted in solvent for analysis, detection levels would be raised above the TC limits. Therefore, analysis of samples would provide results that would not be useful for comparison to regulatory criteria. The proposed approach will minimize the exposure of sampling and analytical personnel and provide a conservative basis for waste designation.

Both radiological limitations and analytical/matrix limitations will affect the analysis of samples for waste designation, as discussed below:

- The 222-S Laboratory, where any analysis of highly radioactive samples would be conducted, has a hood limit of 0.024 Curie (Ci), which is equivalent to about 0.4 g of Pu. If samples are estimated to contain a maximum Pu concentration of 25%, then the maximum test portion that can be handled in the hood is about 1.5 g. The health physics staff at the 222-S Laboratory, however, have indicated that, based on previous experience with high Pu samples, they may require a reduction of the hood limit to 0.01 Ci, which would be equivalent to a test portion of about 0.6 g.
- Given the 50 ppm action level for PCBs, it should be possible to analyze low viscosity oils for total PCBs as Aroclors with attainment of desired method detection and quantitation limits. The laboratory will analyze direct dilutions of liquid oil by gas chromatography (GC).

Determination of pH for the Sludge sample should not present any difficulty and can be performed in the glovebox at PFP.

#### 1.2.3.3 General Analytical Method Information

High radiation levels can present problems with the analysis for organic and inorganic constituents in a waste matrix. Because the Group 2b items contain significant quantities of Pu, the materials must be managed appropriately to minimize exposure of sampling and analytical personnel. The concentrations of Pu in the matrix for a given item will determine the appropriate steps to manage personnel exposure and to establish the appropriate analytical parameters for a given sample. As noted above, several items that were removed from the vault for stability evaluation presented high exposure rates to the laboratory personnel involved with their evaluation (Ewalt 2001). These items are considered to be representative of the materials in Group 2b.

Background information for the processes that generated the items can be used to help determine the likelihood of regulated constituents in the waste matrix. This information is available in "Plutonium Alloys Report" (Borisch 2000), as well as other documents reviewed during preparation of this DQO. The resulting list of potential contaminants would normally be compared against action levels found in the Washington State and Environmental Protection Agency (EPA) regulations for dangerous/hazardous waste. A sampling strategy would then be prepared to determine the best methods for analyzing the waste matrix. Because of the concern for personnel dose rates that would accompany sampling and analysis of the Group 2b items for the full range of COPCs, the items will be assigned waste codes based primarily on process knowledge, rather than through analyses. The following logic will be applied for the various decision statements:

1. The presence of TC VOCs will be stipulated for any items in which there is evidence that oils are present in a low-viscosity form (i.e., the oil is in a liquid state).

2. Because process knowledge and the limited available data suggest the potential presence of TC metals in the alloys, their presence will be stipulated for all items.
3. A qualitative analysis for reactivity (i.e., visibly looking for a reaction when exposed to air) will be conducted on all items as their cans are opened, to supplement information generated through management history and limited bench-scale observations.
4. For the Turnings in Oil, an attempt will be made to collect a sample for laboratory analysis of PCBs. If there is sufficient low viscosity, free oil present in the Sludge or Sweeps, a sample will be collected for laboratory analysis for PCBs.
5. Sludge will be sampled and analyzed for pH to evaluate for the characteristic of corrosivity.

Inputs for Decision Statements 1, 2, and 3 are based on process knowledge and a visual examination of the items. No analyses will be performed to support designation for these criteria.

Decision Statement 4 requires an evaluation of analytical results against the TSCA limit for PCBs as Aroclors, which is equal to 50 ppm. In order to analyze for PCBs, the oil must first be dissolved in a solvent. The limited evaluation of the Turnings in Oil suggests that it may be difficult to obtain a sample of oil to analyze for PCBs. Therefore, sampling and analysis should be conducted for PCBs only if there is sufficient free oil available to perform the analysis (1 g).

PCB sample preparation should be performed by SW-846 Method 3580A (Waste Dilution), followed by analysis using Method 8082 (GC/Electron Conductivity Detector). The two samples from the Turnings in Oil item that was removed from storage for stability evaluation contain approximately 1/30 mL and 1/10 mL of oil in the bottom of a 60 mL glass bottle. Although this quantity is not sufficient for laboratory analysis, it can provide a qualitative basis for evaluating the oils for potential for PCBs. As noted elsewhere in this document, there is a strong likelihood that the oil used to store the turnings contained Lard Oil. Lard Oil was used as a machine oil for Pu because of its high natural tolerance for heat. This characteristic of the oil argues against a need to add PCBs. Alternatively, the oil could be a petroleum-based oil, which might have contained PCBs for their heat-resistant qualities.

Figure 1 shows a logic that is being used to evaluate the oils for the presence of PCBs. In order to analyze the oil for PCBs, a sample must first be dissolved in either methanol or hexane. Lard Oil contains polar and non-polar constituents, but contains primarily high molecular weight polar fatty acids. Polar constituents will be soluble in methanol and non-polar constituents will be soluble in hexane. A petroleum-based oil will be non-polar and, thus, dissolve in hexane. While the solubility test is not 100% conclusive, a more polar material generally will dissolve in methanol and a less polar or non-polar will dissolve in hexane. EPA SW-846 methods (e.g., 3510, 8081, 8082) use hexane to dissolve hydrocarbon oils and waste that contain PCBs. Hexane is used because hydrocarbons such as transformer oil and PCBs are soluble due to their less polar nature.

The small quantities of oil from the examination of the initial two items discussed in the previous paragraph were used to evaluate solubility in methanol and hexane. This activity is the preliminary determination from the opportunistic samples (Steps A and B), shown in Figure 1. The following logic was established, based on solubility: if the oil dissolved, an attempt would be made to collect a sufficient volume of oil from the remaining Turnings in Oil items to meet analytical requirements (1 g).

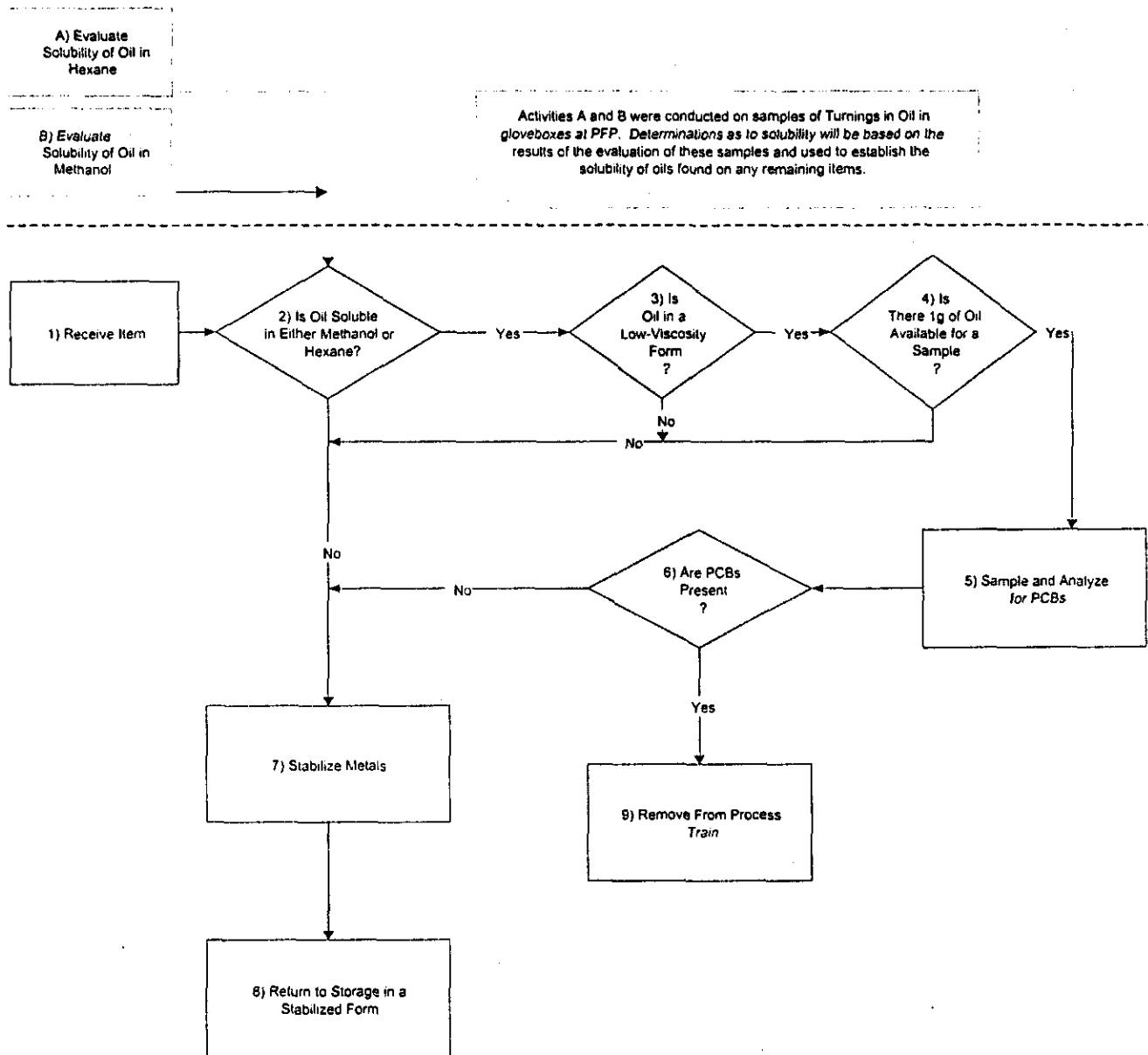
- If the oil did not dissolve, the assumption would be made that, due to low solubility, the oil is Lard Oil and unlikely to contain PCBs; no further attempts at analysis for PCBs would be performed for the Turnings in Oil.
- If the oil dissolved, but there is not adequate oil available to make up a sample, or the oil is of high viscosity and cannot be removed from the turnings, the oil will not be analyzed. This result is based on the need for at least 1 g (or mL) of material so that quality control (QC) can be performed and detection limits can be achieved.

A preliminary solubility evaluation of the oil through mixing with hexane and methanol showed the oil to be soluble in methanol, but not hexane (Cooper 2001). Since the oil from the turnings was insoluble in hexane, this result provides supporting evidence that the oil is polar and does not contain PCBs. Without performing infra-red analysis on the oil or full-blown method development, this solubility test is the best approach short of full analysis with the few milliliters of sample that were present. An attempt will now be made to collect 1 g of oil to perform the analysis.

Oils are not anticipated to be present in any of the items other than the Turnings in Oil. If pourable oil is found in any other items, one sample will be collected for each subgroup that contains oil. This sample should be collected from one item, if possible. If sufficient oil cannot be collected from one item, oil will be added from additional items, if available, to meet the analytical needs (1 g). No more than one sample will be collected from any subgroup.

Decision Statement #5 requires analysis of the Sludge for pH. The Sludge will be considered as corrosive if the pH is less than 2 or greater than 12.5. Determination of pH can be performed using narrow-range pH paper.

Figure 1. Logic for Sampling Group 2b Oils for PCBs



## 1.2.3.4 Summary of COPCs

Table 4 lists the TC constituents from 40 CFR 261.24/WAC 173-303-090(8) and identifies those that will be stipulated as present, retained for analysis, or eliminated from additional consideration, as well as the basis for eliminating compounds from further consideration. The duration and nature of storage for the alloy materials would effectively eliminate any of the VOCs that might have been present. Nonetheless, because volatile organic analysis could not be performed within holding times to confirm that VOCs are not present, and because of the issues associated with solvent dilution of oil, items that potentially contain VOC constituents (i.e., any items with oil) will be designated for the compounds identified in Table 4. Adding the VOC waste codes will not alter how the waste is managed. Semi-volatile compounds were eliminated from further consideration because there is no basis for believing that these compounds were used (Appendix 2).

Table 4. Summary Disposition of COPCs for Group 2b Alloys (2 pages)

Waste Number	Constituent	Action			Basis for Elimination				
		Stipulate	Analysis	Eliminated	Not used in metallurgy or fabrication	VOC	SVOC	Pesticides	Explosive
D004	Arsenic	X							
D005	Barium	X							
D018	Benzene			X		X			
D006	Cadmium	X							
D019	Carbon tetrachloride	X**				X			
D020	Chlordane			X	X			X	
D021	Chlorobenzene			X	X	X			
D022	Chloroform			X	X	X			
D007	Chromium	X							
D023	o-Cresol			X	X		X		
D024	m-Cresol			X	X		X		
D025	p-Cresol			X	X		X		
D026	Cresol			X	X		X		
D016	2,4-D (Dichlorophenoxy acetic acid)			X	X			X	
D027	1,4-Dichlorobenzene			X	X	X		X	
D028	1,2-Dichloroethane			X	X	X			
D029	1,1-Dichloroethylene			X	X	X			
D030	2,4-Dinitrotoluene			X			X		X
D012	Endrin			X	X			X	
D031	Heptachlor and its epoxides			X	X			X	
D032	Hexachlorobenzene			X	X		X		
D033	Hexachlorobutadiene	X**			X		X		
D034	Hexachloroethane			X			X		
D008	Lead	X							
D013	Lindane			X	X			X	

Table 4. Summary Disposition of COPCs for Group 2b Alloys (2 pages)

Waste Number	Constituent	Action			Basis for Elimination				
		Stipulate	Analysis	Eliminated	Not used in metallurgy or fabrication	VOC	SVOC	Pesticides	Explosive
D009	Mercury	X*							
D014	Methoxychlor			X	X			X	
D035	Methyl ethyl ketone			X		X			
D036	Nitrobenzene			X			X		
D037	Pentachlorophenol			X	X			X	
D038	Pyridine			X			X		X
D010	Selenium			X					
D011	Silver	X							
D039	Tetrachloroethylene	X**				X			
D015	Toxaphene			X				X	
D040	Trichloroethylene			X		X			
D041	2,4,5-Trichlorophenol			X	X			X	
D042	2,4,6-Trichlorophenol			X	X			X	
D017	2,4,5-TP (Silvex)			X	X			X	
D043	Vinyl chloride	X**				X			
-	Polychlorinated biphenyls as Aroclors		X***						

\* Possible contaminant in Sweeps only due to possibility of broken thermometer.

\*\* If pourable oils are present, these constituents will be stipulated as present in the relevant waste forms rather than performing sampling and analysis.

\*\*\* PCB analysis will be performed only if there are pourable oils present.

#### 1.2.4 Step 4 – Define Boundaries

This DQO process and results apply to the decisions required to support designation of the 38 items listed for Group 2b. In order to minimize exposure of sampling and analytical personnel, any results from PCB analysis of the Turnings in Oil sample will be applied to all items in that subgroup. Table 5 indicates the approach for evaluating the various waste constituent concerns.

**Table 5. Sampling Approach (1 page)**

Group 2b Subgroup	Stipulate for VOCs?	Stipulate TC Metals <sup>2</sup> ?	Analyze PCBs?
Turnings in Oil	Yes <sup>1</sup>	Yes	Yes <sup>5</sup>
Skulls, Chips, and Turnings	NA	Yes	NA
Not Specified Pu/Al Scrap	NA	Yes	NA
Pu/Zr Alloy	NA	Yes	NA
Sludge <sup>6</sup>	Yes <sup>1</sup>	Yes	Yes <sup>5</sup>
Sweeps	Yes <sup>1</sup>	Yes <sup>3</sup>	Yes <sup>5</sup>
Plastic Mounts	NA	Yes <sup>4</sup>	NA

1. If the oil in the Turnings in Oil is in a low-viscosity (liquid or semi-liquid) state, VOC constituents will be stipulated as present. If the sludge or sweeps appears oily, it will be designated for VOC constituents.
  2. TC metals, with the exception of mercury, will be stipulated as present in all items.
  3. Mercury will be added for Sweeps only.
  4. TC Metals will be stipulated for the Plastic mount on the assumption that there is alloy present in the mount.
  5. Analyzed only if an oil sample can be collected.
  6. Sludge will be analyzed for corrosivity (pH).
- NA – Indicates that this analysis is not considered to be relevant for the subgroup items.

### 1.2.5 Step 5 – Decision Rule

Decisions will be made on an iterative basis, based on the sequential analyses that are performed for each subgroup. If analysis is performed (i.e., for PCBs), the results of one sample from each subgroup will be used to support the designation for that subgroup. The decision rules in Table 6 are numbered to maintain the relationship to the decisions listed in Step 2.

DR#	Table 6. Decision Rules (2 pages)
1A	If observations of [Oil/Sludge/Sweeps] indicate that the oils are present in a low-viscosity (liquid or semi-liquid) condition, then the items in the relevant Subgroup(s) will be managed as TRU-mixed waste and TC VOCs will be stipulated as present.
1B	If observations of [Oil/Sludge/Sweeps] indicate that the oils are present in a high-viscosity/solidified state, then the items in the relevant Subgroup(s) will be managed as TRU-only waste, in the absence of any other regulated constituents of concern. <sup>1</sup>
2	Based on process knowledge and to minimize exposure of sampling and analytical personnel, all items will be stipulated as containing TC metals, except mercury. Because of the potential for a broken thermometer in a hot cell, mercury also will be stipulated as present in the sweeps.
3A	If process knowledge and observation indicate a reactivity potential for a specific item when opened, <i>based on a qualitative observation of its reaction when exposed to air</i> , the item will be managed for the characteristic of reactivity as TRU-mixed waste.
3B	If process knowledge and observations indicate the specific items in a container being opened do not possess the characteristic of reactivity, <i>based on a qualitative observation of their reaction when exposed to air</i> , the items will be managed as TRU-only waste, in the absence of any other regulated constituents of concern. <sup>1</sup>

DR#	Table 6. Decision Rules (2 pages)
4A	If analysis of samples from the [Turnings in Oil/Sludge/Sweeps] for PCBs are above the regulatory action level (50 ppm), then the items in the relevant Subgroup(s) will be managed as TRU-mixed (TSCA) waste and evaluated for the presence of any other regulated constituents of concern.
4B	If analysis of samples from the [Turnings in Oil/Sludge/Sweeps] for PCBs are below the regulatory action level (50 ppm) or an insufficient sample can be collected for analysis, then the items in the relevant Subgroup(s) will be managed as TRU-only waste, in the absence of any other regulated constituents of concern. <sup>1</sup>
5A	If the pH of the sludge is less than 2 or greater than 12.5, the Sludge will be designated for the characteristic of corrosivity and managed as a TRU-mixed waste.
5B	If the pH of the Sludge is between 2 and 12.5, then the sludge will be managed as TRU-only waste, in the absence of other regulated constituents of concern. <sup>1</sup>

1. n.b., All items will be designated as mixed waste, based on the stipulation from DR #2 that all items contain TC metals.

### 1.2.6 Step 6 -Specify Tolerable Limits

Note that the sampling is not statistically based. The primary reason is that the condition (i.e., size) and physical state (i.e., liquid or solidified oil) of the materials will determine which containers can be sampled. The status cannot be determined until the containers are opened and examined. Due to ALARA concerns, FH plans to minimize the handling and only open containers once. In addition, for the Sludge and Sweeps subgroups, there is only one container in each subgroup. Finally, the fact that these items were all generated through essentially the same physical process, coupled with the ALARA concerns associated with sampling and analysis, argues in favor of limited sampling with the results applied to the group as a whole. This approach is consistent with the NRC/EPA Policy on Sampling Mixed Wastes (NRC 1997).

### 1.2.7 Step 7 - Optimize the Design

Oil was used to protect solids from exposure to air and moisture. It has been observed that, for some items, the oil has solidified to the consistency of dried shellac; therefore, oil may range from a high viscosity liquid to a low viscosity liquid or, potentially, a semisolid or solid material. A sample of oil will be obtained for analysis of PCBs only when the oil is pourable or scrapable and 1 g of sample can be collected. When oil is solidified or semisolid, an attempt will be made to collect a sample for PCB analysis by scraping a sample into a jar. However, scraping of metals that have sharp edges (e.g., chips and turnings) will not be done, as these could puncture a glove and result in direct exposure of the individual performing the sampling.

Samples will potentially be collected from the following subgroups for the specified analyses, based on the logic presented in the preceding sections:

1. Turnings in Oil – oil for PCBs, if present
2. Sludge – for pH; oil for PCBs, if present
3. Sweeps –oil for PCBs, if present

In order to avoid potentially oxidizing Pu in the waste, material will not be stirred or mixed before sampling. If a sample cannot be collected, the oil will be assumed to be Lard Oil and PCBs are assumed as not present.

Because analysis for metals would require a high dose rate to personnel, TC metals (with the exception of mercury) will be stipulated for all items in lieu of sampling. Because broken thermometers are relatively common in gloveboxes, there is a potential concern over the presence of mercury in the Sweeps, which originated from a glovebox. Therefore, mercury will be added to the list of TC metals for the Sweeps. There is no basis to suspect its presence in the other items.

Because the "Not Specified Scrap" group is expected to consist of Pu/Al materials that are consistent with the groups listed above, no samples will be collected from this group, unless pourable oils are present. These items will be designated based on the results of evaluation from the other Pu/Al subgroups. If upon opening, the items in a container do not appear to be consistent with the other Group 2b items (e.g., solid, plate-like material, metal other than Pu/Al or Pu/Zr), the container should be set aside or returned to the vault pending development of a strategy to evaluate its contents.

The Pu/Zr items are believed to be large pieces and, because the fabrication process would have been similar, the materials should be consistent with those in the Pu/Al groups.

The Plastic Mount will not be sampled because it is expected to consist of solidified plastic used to mount samples for metallurgical analysis. Plastic is not normally considered as RCRA Dangerous Waste and, in fact, is routinely used in some forms to package waste materials. The plastic is expected to contain some of the metals mounted for analysis; therefore, it will be designated for metals. No other waste codes will be assigned to this item.

A summary of the logic for evaluation and waste characterization of the Group 2b items is provided below:

1. Turnings in Oil

- The contents of the containers will be evaluated qualitatively for reactivity when each item is opened.
- An attempt will be made to collect a sufficient sample (1 g) of oil for PCB analysis if pourable oil is present, provided that obtaining the sample could not potentially rip open a glove. The oil sample will be analyzed for total PCBs as Aroclors.
- If the oil is in a low-viscosity form (i.e., liquid or semisolid), the oil will be designated for TC volatile organic constituents. No analysis will be performed for volatile organics.
- TC metals, with the exception of mercury, will be stipulated as present for all items in this subgroup.

**2. Skulls, Chips, and Turnings**

- All items in the subgroup will be evaluated qualitatively for reactivity as the containers are opened for repackaging.
- TC metals, with the exception of mercury, will be stipulated as present for all items in this subgroup.
- If any oil is present and pourable, it will be analyzed for total PCBs as Aroclors. (Note that no oil is expected in the Skulls, Chips, and Turnings subgroup.) If the oil is in a low-viscosity form, it will be stipulated as containing TC VOCs.

**3. Sludge**

- The sludge is assumed to be dried material. It is possible that the sludge may be a solidified monolith of sodium hydroxide (NaOH) sludge. The sludge will be analyzed for total PCBs as Aroclors if free-flowing oil is present.
- If low-viscosity oils are present, the sludge will be designated for TC VOCs.
- TC metals will be stipulated as present in the sludge.
- A sample of the sludge will be collected for pH determination using narrow-range pH paper.

**4. Sweeps**

- The sweeps are expected to be dried material. TC metals, including mercury, will be stipulated as present.
- Any paper, cloth, or plastic debris will be segregated for disposition through an alternate pathway.
- As with the Sludge, if free oil is present, a sample will be collected and analyzed for total PCBs as Aroclors.

**5. Pu/Zr Metals and the "Not Specified Items" should be similar in composition to the "Skulls, Chips, and Turnings" items and will be evaluated through the same procedures as these items. All items in these groups will be stipulated as containing the TC metals (except mercury).****6. Plastic Mount – this item will be evaluated qualitatively for the characteristic of reactivity upon opening and stipulated for the presence of TC metals (except mercury).**

All items are assumed to be non-reactive and not ignitable, based on the management history of the alloy materials and the limited observations of those items inspected at PFP.

## 2 SAMPLING AND ANALYSIS PLAN FOR THE PFP GROUP 2B ALLOYS

### 2.1 PURPOSE

The purpose of this Sampling and Analysis Plan (SAP) is to acquire data of known and appropriate quality to characterize the PFP Group 2b Alloys for disposal as waste; i.e., as required by the TSCA and/or the Dangerous Waste Regulations (WAC 173-303)/RCRA, as appropriate. The material is known to be TRU and is destined for disposition to the WIPP near Carlsbad, New Mexico.

The parameters to be addressed by the SAP are (1) the total concentration of PCBs as Aroclors in any pourable oil that may be associated with the Group 2b Turnings in Oil, sludge, sweeps, or any other pourable oil, and (2) the pH of the Group 2b Sludge.

The Action Limit for PCBs as Aroclors is 50 ppm. Because this action limit is based upon a summation of the various Aroclors, the method detection limit (MDL) for each Aroclor should be no greater than approximately 5 ppm.

The Action Limit for the pH determination is related to the definition of corrosivity in the Dangerous Waste Regulations (Washington Administrative Code 173-303-090) and the Code of Federal Regulations, 40 CFR 261.22. A waste is corrosive if the pH is less than or equal to 2 or greater than or equal to 12.5.

### 2.2 CRITERIA FOR SAMPLING

A sample will be taken from the Turnings in Oil group for analysis of PCBs as Aroclors only if the material containers have free oil in a pourable state; that is, the oil must be of low enough viscosity to be easily separated from the metal pieces and fines and 1 mL of sample is available. A 1 mL sample is needed to perform the analysis and the associated QC and achieve the applicable laboratory reporting limits. The same criteria apply to the collection of oil from any other subgroups. Oil may be collected from more than one item in a subgroup if necessary to gather the required volume for analysis. Only one sample is required to be collected for each subgroup. If a single item contains a sufficient volume of oil to collect two, 1-mL (minimum size) samples, two samples should be collected.

A sample will be taken from the Sludge group for determination of pH only if fine material is available or can be easily obtained as the Sludge is manipulated within the glovebox. Anecdotal reports indicate that the sludge may be a dense monolith that cannot be crushed or broken to obtain a sample, in which case a sample will not be obtained.

### 2.3 SAMPLING AND ANALYSIS

*Caution: Usually samples are mixed before sampling. Because mixing may promote oxidation and thereby generate heat, mixing is not to be done before sampling. This is a safety issue.*

## 2.3.1 Oil

### 2.3.1.1 Sampling

Complete the label of the sample vials before sampling is performed. Include at least the following information in addition to the unique sample number assigned by the 222-S Laboratory:

- original Turnings in Oil container number or identity,
- date and time sample taken, and
- name of sampler.

All other information can be entered in the logbook and on the chain-of-custody form if there is not enough room on the label.

Pour or pipet at least 1 mL of oil from the Turnings in Oil container(s). If possible, place the oil directly into the clean, 60 mL glass vial and cap with the Teflon lid. Oil from different Turnings in Oil containers may be combined in one vial if necessary to achieve the 1 mL minimum sample size. If sufficient oil is available to obtain individual samples of at least 1 mL from each container, obtain at least two distinct samples.

*Note 1: The 60 mL glass vials with Teflon lids are supplied by Project Hanford Management Contract (PHMC) Analytical Services. Have several extra on hand in case of breakage. Other sizes of glass vials may be used, provided they are new, never used, and have Teflon lids or lid inserts.*

*Note 2: The oil sample, internal surfaces of the pipette and glass vial, and the Teflon lid must not come into contact with plastics because plasticizer contamination will detrimentally affect the PCB analysis.*

The designated Sample Custodian must complete the chain-of-custody documentation as required by the Analytical Services procedure, "Chain-of-Custody for Environmental Media and Waste Samples," ASP-200. Use as many custody forms as needed and note the number of pages in the top right corner. The total number of pages of the chain-of-custody documentation must be recorded on the forms as "page x of y" where "x" is the sequential page number and "y" is the total number of pages. The Sample Custodian must sign the Chain-of-Custody form, as he/she is responsible for the samples, and must indicate on the documentation if the samples are placed in the vault before transfer to the 222-S Laboratory. Each time the samples are relinquished to another person or a secure location, the transfer must be documented and signed on the Chain-of-Custody form as specified in the ASP-200 procedure.

Table 7 describes how to complete the chain-of-custody documentation:

**Table 7. Chain of Custody Documentation (1 page)**

<b>Form Title</b>	<b>Information to be added</b>
Collector	Name of person(s) collecting the samples
Company contact and phone	Name and phone of person to contact regarding sampling process
Project coordinator	Leave blank
Price code, air quality Data turnaround	Leave blank for now
Project Designation	Group 2b Alloys - PCBs as Aroclors
Sampling location	Building and room where samples were collected
SAF no.	Leave blank
Ice Chest	Does not apply, leave blank
Field log book no.	Enter the logbook number in which sampling information is recorded
COA	Leave blank
Method of shipment	Leave blank
Shipped to	Leave blank
Offsite property	Leave blank
Bill of Lading	Leave Blank
Possible sample hazards	Oil may contain fine plutonium/aluminum alloys. These may oxidize and generate heat or flame.
Preservation	None
Type of container	G = glass
No. Of containers	Enter #
Volume	Enter volume
Sample No.	Enter number that corresponds to the drum/container of original sample. <b>Double check - this is very important!</b>
Matrix	Oil
Sample date	Enter date sampled
Sample time	Enter time sampled
Sample	Place check marks under applicable analysis: PCBs for oil
Sign relinquished by or received by	Signature, as applicable. Must be legible!
Special instructions	Fine metal pieces may be included in the oil sample. Note that these are plutonium/aluminum alloys, which may generate heat if removed from the oil.
Laboratory and final sample disposition	Leave blank

Any samples taken will be transferred to the PFP Analytical Laboratory and prepared, then held, for shipment until such time that they are transported to the 222-S Laboratory for analysis. The sampler will sign the Chain-of-Custody form to show the custody is relinquished to the appropriate responsible individual, who will sign to document acceptance of the samples.

Immediately after the oil samples are collected and chain-of-custody documentation is completed, provide copies of the logbook pages and chain-of-custody documentation to the data quality assessment contractor (FAX to Environmental Quality Management at 509-946-4595) so the documentation can be evaluated for legibility, traceability, and completeness.

When the sample(s) are submitted to the 222-S Laboratory, a Sample Analysis Request form must be completed. This form and the instructions for it can be obtained via FAX from Kathy Powell, Analytical Project Manager, phone 372-0939 or cell phone 521-0320, pager 85-7104.

#### **2.3.1.2 Holding Time and Preservation**

The sample(s) of oil will be transported to the 222-S Laboratory as soon as possible, given constraints such as radioactive material inventory within the 222-S Laboratory, if applicable to the oil sample, and project budget. Holding time and preservation shall not be a concern for the oil sample because PCBs are known to be persistent in the environment and stable at high temperatures. However, the holding time and preservation requirements of the analytical method shall apply to the extract of the oil that is to be analyzed by GC (e.g., 40 days from an extraction to analysis, extracts kept under refrigeration).

#### **2.3.1.3 Analysis**

The 222-S Laboratory shall analyze the oil sample for PCBs as Aroclors according to SW-846 Method 8082. Any preparative cleanup of the sample shall be at the discretion of the laboratory, with the requirement that the procedure be from the latest version of SW-846, as implemented by the Laboratory's procedures. Both confirmation and quantitation are required for any Aroclors identified.

Because the regulatory Action Limit of 50 ppm is based upon a summation of the various Aroclors, the MDL for each Aroclor should be no greater than approximately 5 ppm. Accuracy, as measured on the laboratory control sample (LCS) (as percent recovery), shall be from 70 to 130%, and on the matrix spike samples (as percent recovery) shall be from 75 to 125%. Precision as measured by the relative percent difference of duplicate or matrix spike duplicate samples shall be less than 25%.

Other requirements, such as surrogate recovery, shall be according to the requirements of the SW-846 method.

Data shall be reported as a preliminary summary report and as a full data package suitable for complete validation. The project also requires an electronic deliverable. See the quality assurance (QA) project plan (Section 3) for additional information.

## 2.3.2 Sludge

### 2.3.2.1 Sampling

Place fine particles or easily crushed pieces of the Sludge material into two (or preferably four) clean, disposable glass containers, if the pH determination is to be done immediately after sampling. Document the appearance and approximate amount available for the pH determination in the controlled logbook.

*Note: If the pH determination is to be done at a later time, the sample material must be placed in a glass vial and all requirements described above (Section 2.3.1.1) will apply for the Sludge, as well.*

### 2.3.2.2 Holding Time and Preservation

Although no specific holding time and preservation requirements apply, the Sludge material should be held for only a short period of time before the pH determination is performed. If the material was crushed, newly exposed surfaces can oxidize over time and potentially affect the pH measurement.

### 2.3.2.3 Analysis

Both the SW-846 Method 9045C (EPA 1997) and the American Society for Testing and Materials (ASTM) Method D4980-89 (ASTM 1989) involve mixing the test portion with an equal amount of reagent water. The ASTM method allows the use of pH paper as opposed to the SW-846 method that uses a pH meter. The use of pH paper is recommended to minimize exposure to radiation. After a short waiting period, the liquid is placed on pH test paper with a clean glass rod, tube, or dropper. (Do not dip the pH paper into the liquid! Erroneous data will be the result!) Wide-range pH paper may be used to identify the pH to within about 1 pH unit. If necessary (i.e., the initial pH measurement is within 1 pH unit of 2 or 12.5), a narrow-range pH paper is then used to make a more accurate determination. If sufficient sample material is available, perform a duplicate of the "final" definitive pH determination.

For QC, the wide range pH paper should be tested on two buffers near the Action Limits of pH 2 and pH 12.5 (buffers of pH 2 and pH 12 are recommended). This test can be performed outside the glovebox, but should be performed by the same analyst on the same day as the routine sample(s) are analyzed in the glovebox. Ensure the buffers are fresh and have not exceeded their expiration dates. Note that only the amount of pH paper required for the pH determination needs to be introduced to the glovebox; the paper may be torn from the dispenser roll and placed in a zip-lock plastic bag for transfer to the glovebox. However, the appropriate color chart will be required to be placed into the glovebox so that the pH paper can be read against the colors resulting at the various pH levels.

For project records, record the following in the controlled logbook:

1. Description of method used for analysis (i.e., method number and any modifications);
2. Identity of original container from which sample material was taken;
3. Amount of sample mixed with water;
4. Results of the pH tests, date and time of determination, and name of analyst;
5. Range of pH for the test papers used, lot numbers, and name of manufacturer;
6. Theoretical pH of each of the check buffers used with expiration date and the name of the respective manufacturer.

Immediately after the logbook entries are completed, provide copies of the logbook pages to the data quality assessment contractor (FAX to Environmental Quality Management at 509-946-4595) so the documentation can be evaluated for legibility, traceability, and completeness.

### **3 QA PROJECT PLAN FOR THE PFP GROUP 2B ALLOY MATERIALS**

This section includes descriptions of plans and programs to assure that the quality of the information generated by this SAP is consistent with the requirements for sampling and analysis contained in SW-846, Chapter 1. It includes the following topics: project management, training, quality objectives and criteria for measurement data, data acquisition, data reporting, data review and validation, and data quality assessment.

#### **3.1 PROJECT MANAGEMENT**

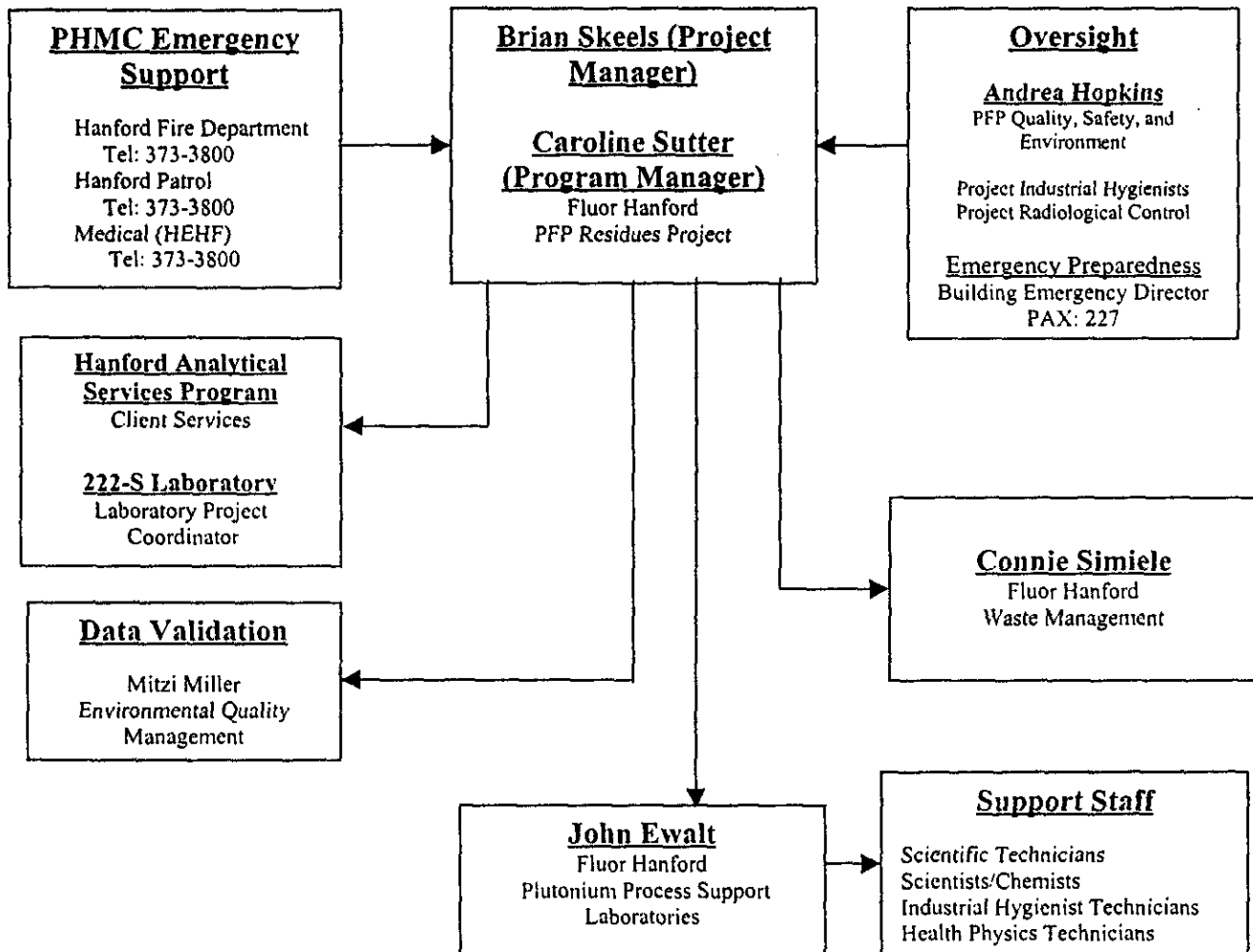
Figure 2 provides the organization chart for activities associated with sampling and data acquisition for characterization of the PFP Group 2b Alloy materials. The Fluor Hanford PFP Residues Project, supported by the PPSL and PFP Analytical Laboratory, has responsibility for implementation of the sampling activities and determination of pH on the Sludge. Duratek Federal Services of Hanford (222-S) is responsible for laboratory data acquisition, and reporting. Environmental Quality Management will perform data validation.

All planned work at PFP will be reviewed and released per FSP-PFP-5-8 within the existing work control system. All work planning and performance at PFP must be within the existing safety authorization basis.

The safety basis and work authorization will be maintained by Fluor Hanford for all work associated with sampling at PFP and characterization of the Sludge for pH. All work will be conducted utilizing the controls identified in the SAP and in the specific test plan developed per FSP-PFP-5-8.

Before initiating activities related to the sampling and characterization at PFP, a contractor Standard Startup Review will be held as required by HNF-PRO-055 to ensure all prerequisites have been met and all assigned organizations and individuals are adequately trained and prepared for their assigned tasks.

Figure 2. Organization Chart



### 3.1.1 Emergency Preparedness/Response

This work is subject to the appropriate building contingency plan (either for PFP or 222-S Laboratory, as appropriate), which incorporates existing emergency procedures as part of the site-specific health and safety program.

It is expected that all workers are current in their facility orientation training and emergency response actions. Fluor Hanford maintains the official training records.

### 3.1.2 Industrial Safety and Health

Fluor Hanford is responsible for job-specific health and safety planning for the sampling activities, as required by the Integrated Safety Management System (Integrated Environmental, Safety, and Health Management System Description, HNF-MP-003, Rev. 4). Job hazards analyses will be performed and documented as required by the organizations responsible for specific tasks.

### 3.1.3 Laboratory Analysis

Fluor Hanford will provide overall project planning and control of all laboratory analysis requests.

The Hanford Analytical Services Program, Client Services, and the 222-S Laboratory (*Duratek Federal Services of Hanford, Inc.*) will provide or coordinate analytical laboratory support. This support includes assurance that all procedures used meet the requirements of the DQO as well as those of the *Hanford Analytical Services Quality Assurance Requirements Documents* (HASQARD) (DOE-RL 1998).

### 3.1.4 Nuclear Safety

Fluor Hanford will prepare or coordinate the preparation of any necessary criticality analysis. Any special nuclear safety requirements, beyond the standard radiological control requirements, will be defined *in the work plan or procedure*. At PFP, the Residues Project Manager and PFP Project Manager are responsible to assure that all procedures and work plans are consistent and that all work complies strictly with all applicable nuclear safety requirements. At the 222-S Laboratory, the Laboratory Project Coordinator, is responsible for adherence to all work package and nuclear safety requirements.

### 3.1.5 Operations

All operations staff at PFP will be under the direction and control of the Residues Project Manager or Team Leads, who will conduct the pre-job briefing(s). PFP Operations staff will ensure that all necessary equipment for sampling and pH characterization at PFP is obtained in advance of the work.

### **3.1.6 Program Management**

Caroline Sutter will have overall programmatic responsibility. This includes preparing any change requests and special presentations. The 222-S Laboratory will designate a project manager to assist in management of the analytical laboratory portion of this work. The PFP or 222-S Laboratory management has the lead in defining and implementing all readiness review actions required in the respective facilities before implementing this work. This includes scheduling necessary plant review committee meetings to review final work packages.

### **3.1.7 Radiation Control**

Fluor Hanford will perform surveys of radiation levels according to requirements established in the radiation work package and in governing requirements of PFP and the 222-S Laboratory. In order to keep exposure of personnel ALARA, personnel access to the project areas will be limited. Observers may be allowed, but not encouraged to be present at the work locations because of high dose rates from the materials.

### **3.1.8 Environmental Regulation**

Fluor Hanford is responsible for any environmental approvals that may be needed to support the work at PFP or at the 222-S Laboratory. Duratek Federal Services of Hanford, Inc. will support this effort via document reviews, and providing process descriptions or other information on work techniques, as requested. Fluor Hanford, through the DOE, Richland Operations Office (RL), is responsible for formal release of the SAP to State or federal regulators. The laboratory staff will not release information to DOE or the regulators without prior approval from PFP Program Management.

### **3.1.9 Laboratory Services/Data Validation**

The laboratory project coordinator will serve as the single point-of-contact for all work conducted within the 222-S Laboratory. Environmental Quality Management will receive the data packages from the Laboratory Project Coordinator to perform the data validation.

### **3.1.10 Scheduling**

Fluor Hanford support to Caroline Sutter will prepare and maintain a working schedule. The schedule will be reviewed by the PFP Residues Project in a weekly scheduling meeting. The PPSL and the 222-S Laboratory will provide weekly status reports against this schedule. Stated schedules will be provided to Caroline Sutter and/or RL, as requested.

### **3.1.11 Security**

Work will be performed by staff assigned to either PFP or the 222-S Laboratory at their respective locations. Only persons with authorized access and the appropriate badging will be allowed in the work areas, with the exception of others allowed to observe the work if escorted at all times.

### 3.1.12 Training

Fluor Hanford will provide all training necessary to implement the requirements of this plan.

Operations and support staff will have the training necessary to qualify to perform the work (e.g., RadWorker II), including any specialized training for sampling, waste management, or laboratory proficiency.

### 3.1.13 Quality Assurance

QA reviews will be performed at PFP and the 222-S Laboratory, as required, by Fluor Hanford QA staff. QA of the laboratory analysis process, including assuring that the analytical work will meet the requirements of the HASQARD (DOE-RL 1998) and the DQO, is the responsibility of Fluor Hanford, Duratek Federal Services of Hanford, Inc., and the 222-S Laboratory operations personnel.

### 3.1.14 Work Control

All work will be planned and conducted under detailed work procedures and test plans prepared by the responsible organizations. For work at the 222-S Laboratory, work planning and review will be conducted according to requirements established for the Laboratory. Following review, these work packages will be released for implementation.

## 3.2 TRAINING

For PFP and the 222-S Laboratory, each nuclear facility's health and safety requirements ensure that workers have the knowledge and skills necessary to safely execute assigned duties. A graded approach is used to ensure that workers receive a level of training commensurate with their responsibilities, which also complies with applicable DOE orders and government regulations. Specialized employee training includes pre-job briefings, on-the-job training, emergency preparedness, plan of the day, and facility/work site orientations. In addition, all members of the Building Emergency Response Organization receive specialized training. Table 8 presents the training and qualifications applicable for facility work and activities. Facility-specific documentation describes training requirements in greater detail.

Before initiation of activities, Fluor Hanford will conduct a startup review according to the requirements of HNF-PRO-055. This formal review will ensure all work prerequisites have been met and all assigned individuals and organizations are adequately prepared and trained for their assigned tasks.

**Table 8. Radiological Entry Requirements (Summary Table) (1 page)**

<b>Visitors</b>	
All areas.	Must meet requirements of the applicable Radiation Work Permit.
<b>Workers</b>	
The sampling area	1) RadWorker II 2) Task-specific training as delineated in the governing work packages (Training Matrix) and applicable activity hazard analyses.  <i>Pre-Job Safety Plan-of-the-Day briefings including updates on ongoing activities and changing conditions.</i>
Entries into RBA and RA.	24-hr Hazwoper and RadWorker I Training
Entries into CA, HCA, HRA, or ARA.	40-hr Hazwoper and RadWorker II

Note: DOE Facility Representatives may act as the escort for all DOE business and tours.

ARA = airborne radiation area

HRA = high radiation areas

CA = contamination areas

RA = radiation area

HCA = high contamination area

RBA = radiological buffer area

Each employee's training records are maintained and updated, when needed. Current training status for any PHMC employee is accessible via the computer database called the Access Control Entry System (ACES). ACES is used to verify that entry requirements are met for individuals who require access to radiologically controlled areas of the Hanford Site. More detailed information on access control requirements may be found in FSPPFP-5-8.

### 3.3 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The DQO process to support these sampling and analysis activities, described in Section 1, was conducted in accordance with Guidance for the DQO Process (EPA 1994). Input to the DQO process was provided by staff of the following organizations: PFP (engineering and environmental personnel), and Hanford Analytical Services Program Client Services; RL and the Washington State Department of Ecology provided comments to the draft DQO report. The parameters of concern to be addressed by the SAP are (1) the total concentration of PCBs as Aroclors in the pourable oil that may be associated with the Group 2b Alloy Materials and (2) the pH of the Group 2b Sludge.

Sampling activities will be performed using procedures that have been developed for use at the PFP under the conditions existing in the gloveboxes. The procedure for determining pH of the Sludge at PFP is based on the requirements of SW-846 9045C and ASTM D4980-89, (EPA 1997, ASTM D4980). In general, analytical procedures used at the 222-S Laboratory are based on consensus standard methods, adapted for use at the Hanford Site. However, some analytical procedures have been specifically developed for use on highly radioactive samples or for determining parameters specific to high-level radioactive materials.

The QA objective of this plan is to develop implementation guidance that will provide data of known and appropriate quality to manage the PFP Group 2b Alloys for disposal as waste, i.e., as required by the TSCA and/or the Dangerous Waste Regulations (WAC 173-303)/RCRA, as appropriate. The material is known to be TRU and is destined for disposal at the WIPP near Carlsbad, New Mexico. Data quality is assessed by representativeness, comparability, precision, accuracy, and completeness. Definitions of these parameters, applicable guidelines, and level of effort are provided below. The applicable QC guidelines, quantitative target limits, and levels of effort for assessing data quality are dictated by the intended use of the data and the nature of the analytical method.

Representativeness is a measure of how closely the results reflect the actual concentration distribution of the chemical and radiological constituents in the matrix sampled. Sampling plan design, sampling techniques, and sample handling protocols, discussed in other sections of this document, provide documentation to establish that sample identification and integrity are ensured.

Comparability expresses the confidence with which one data set can be compared to another. Data comparability will be maintained using standard procedures, consistent methods, and equivalent units.

Accuracy is an assessment of the closeness of the measured value to the true value. Accuracy of chemical test results in the matrix of interest is normally assessed by spiking samples with known standards and establishing the recovery. A matrix spike is the addition to a sample of known amounts of a standard compound similar to the compounds being measured. Surrogates are compounds spiked in the organic matrix and also are used to assess accuracy. The LCS is used to assess the accuracy of the laboratory processing of a known matrix.

Precision is a measure of the data spread when more than one measurement has been taken on the same sample. Precision can be expressed as the relative percent difference for duplicate measurements. Laboratory duplicates or matrix spike duplicates are included in the project design, enabling estimates of laboratory precision.

Completeness is a measure of the amount of valid data obtained from the analytical measurement process and the complete implementation of defined field procedures. Completeness is calculated as the number of analytical results divided by the number of analyses requested, multiplied by 100. Completeness for this characterization is 100% for pH determination and for PCB analysis.

The estimated quantitation limit (EQL) is the lowest concentration of an analyte that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is determined by methods described in SW-846, Chapter 1 (EPA 1997) and HASQARD (DOQ-RL 1998). EQLs are functions of the analytical method utilized to generate the data and the amount of sample available for analyses. The term EQL is synonymous with the practical quantitation limit (PQL).

### **3.4 MEASUREMENT/DATA ACQUISITION**

Data acquired from QC procedures are used to estimate the quality of analytical data, to determine the need for corrective action in response to identified deficiencies, and to interpret results after corrective action procedures are implemented. Method-specific QC procedures are incorporated in the individual methods.

This section identifies the minimal QC components that should be used in the performance of sampling and analyses, including the QC information that should be documented.

#### **3.4.1 Sample Collection Methods and Requirements**

Pourable oil samples from Group 2b Alloy Materials and samples of Sludge for pH determination shall be collected as described in the SAP.

#### **3.4.2 Sample Management**

All required records pertaining to sample management shall be maintained and updated regularly. These include chain-of-custody forms, sample receipt forms, and sample disposition records. All samples obtained during the course of this project will be controlled from the point of origin to final disposal in accordance with established custody procedures. The laboratory shall provide unique sample identification numbers on the sample containers. The laboratory shall pre-label all sample containers before filling the container. The laboratory records shall allow the correlation of the sample to its source.

For analysis of PCBs, the 222-S Laboratory will provide analytical services that are in accordance with SW-846 or equivalent approved methods. The Laboratory will be informed of the sampling schedule. The Laboratory Project Coordinator will assure that analyses are performed and records include the location of analysis and the person performing the analysis.

The sample(s) of oil will be transported to the 222-S Laboratory as soon as possible, given constraints such as radioactive material inventory within the 222-S Laboratory, if applicable to the oil sample, and project budget. Holding time and preservation shall not be a concern for the oil sample, because PCBs are known to be persistent in the environment and stable at high temperatures. However, the holding time and preservation requirements of the analytical method (40 days) shall apply to the extract of the oil that is to be analyzed by GC.

The sample for pH determination will be collected in the PFP glovebox and transferred to the PFP Analytical Laboratory for analysis; therefore, some of the sample management practices described above will not apply. If the pH determination is made on the day the sample is obtained and the sample has not been removed from the sampler's custody, sample containers, labeling, and chain-of-custody documentation are not required. The records for the pH determination shall include, but are not limited to the following: results of the determination, QC information (described in the SAP), correlation of the sample to its source, name of the sampler/analyst, date of the activities, and location of the analysis.

### 3.4.3 Sampling Quality Control

Trip blank and field blank samples will not be collected for the oil analysis. PCBs would not be used in processes that could lead to false results. QC applicable to this sampling activity includes collection of a sample duplicate, use of cleaned glass containers for the oil sample, avoidance of plastics in the collection activities (because the plasticizers will detrimentally affect the sample for its intended analysis), and the use of appropriate precautions to avoid introducing any contaminants into the sample containers. Because of dose restrictions in transporting the sample to the Laboratory and for managing the sample at the Laboratory, it is desired that no fine metals be introduced into the sample container. QC for the preparation and analysis is described in the SAP in Section 2.3 and in Section 3.4.4 below.

For the pH determination on the Sludge, enough material to perform a duplicate determination in each of the two pH ranges is desirable. QC for the pH determination must include a blank and a duplicate as defined in Section 3.4.4 below.

### 3.4.4 Laboratory Analytical Method Requirements and QC

The analytical requirements are discussed in the SAP. To assure quality measurements, analytical data are obtained with a stringent set of QA samples. These samples and associated requirements are described below:

- One laboratory method blank for each preparation batch is carried through the complete sample preparation and analytical procedure. The results from the analyses are used to assess contamination from reagents, equipment, and the process in the laboratory.
- One LCS or blank spike is performed for every preparation batch of the same matrix for each analytical method to monitor the effectiveness of the sample preparation and analysis process. The results from the analysis are used to assess laboratory performance.
- A matrix spike sample is prepared and analyzed for every matrix or sample preparation batch. An aliquot of the sample is spiked with the analytes of concern. The results of the matrix spike samples are used to document the bias of an analytical process in a given matrix.
- Laboratory duplicates or matrix spike duplicates are used to assess precision and are analyzed at the same frequency as the matrix spike samples. A laboratory duplicate is an aliquot of the same sample, whereas a matrix spike duplicate is a second matrix spike sample of the same sample. To compare two values, the relative percent difference is based on the mean of the two values and is reported as an absolute value. Either a laboratory duplicate or matrix spike duplicate is performed each analytical method.
- The sensitivity or EQL as defined in SW-846, Chapter 1 (EPA 1997), will be determined for PCBs as Aroclors. The EQL is also called the PQL. The EQL must be met in order to assess whether PCBs are below the action limit. If this cannot be done, the PFP Residues Project Manager must be notified immediately.

- The MDL, as defined in SW-846, Chapter 1 (EPA 1997), will be determined on a clean solid matrix for the PCB method to verify that the laboratory can successfully perform this method. This information will be kept on file at the laboratory.
- Both the EQL and MDL must be determined in a manner consistent with Volume 4 of HASQARD (DOE-RL 1998).

Table 9 provides the QC criteria for the oil sample.

**Table 9. QC Criteria for Analysis for PCBs as Aroclors**

Analytical Method	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery	Spike % Recovery	RPD
8082	Gas Chromatography	70-130	75-125	<25

RPD = relative percent difference

### 3.4.5 Quality Control Requirements

#### 3.4.5.1 Quality Control for pH Determination on Sludge

QC measures applicable to the sampling and determination of pH are discussed in the SAP.

#### 3.4.5.2 Quality Control for PCB Analysis of Pourable Oil

The QA program of the 222-S Laboratory is compliant with HASQARD, as described in the 222-S Laboratory QA Plan (Markel 1998). If any other laboratory performs the work described in this SAP, that laboratory shall have an authorized QA plan that complies with HASQARD.

### 3.4.6 Laboratory Instrument/Equipment Testing, Inspection, and Maintenance Requirements

**Operating Procedures.** Laboratory personnel shall follow procedures established in the relevant QA program for testing, inspection, operation and maintenance of all laboratory instruments and equipment. Procedures should be readily available to those performing the task outlined. Any revisions to laboratory procedures should be written, dated, and distributed to all affected individuals to ensure implementation of changes.

**Equipment Maintenance Documentation.** The maintenance record of each system serves as an indication of the adequacy of maintenance schedules and parts inventory. As appropriate, laboratory personnel should follow the maintenance guidelines of the equipment manufacturer. When maintenance is necessary, it should be documented in either standard forms or in logbooks. Maintenance procedures should be clearly defined and written for each measurement system and required support equipment.

### 3.4.7 Laboratory Instrument Calibration Requirements

Calibration is a reproducible reference point to which all sample measurements can be correlated. A sound calibration program should include provisions for documenting frequency, conditions, standards, and records reflecting the calibration history of a measurement system. The accuracy of the calibration standards is important because all data will be in reference to the standards used. A program for verifying and documenting the accuracy and traceability of all working standards against appropriate primary grade standards or the highest quality standards available should be routinely followed. All instrumentation used shall follow established procedures, as specified by methods listed in this SAP and by HASQARD (DOE-RL 1998), for calibration and frequency of maintenance to assure that quality data are obtained during measurements.

Per method 8082, Section 7.4.3.1 and 8000B, the calibration will include a five point mixture of Aroclors 1016 and 1260 evaluated per 8000B criteria for external calibrations. In addition, single point calibrations for the other Aroclors will be performed per Section 7.4.3.2 of method 8082.

### 3.4.8 Modifications, Deviations, Changes, and Observations

Any modifications made to, or deviations from, the prescribed procedures shall be documented in the project notebooks, laboratory reports, and project records in accordance with the QA/QC program and project documents. All such modifications, deviations, and observations will be noted and justified, as appropriate, in the final analytical reports to the project.

Nonconforming sampling and analytical actions or omissions will be identified, controlled, reported, and dispositioned as required by *Nonconforming Item Reporting and Control* (PHMC 1997b).

Method 9045C will be modified to allow the use of narrow-range pH paper or ASTM Method D4980-89 will be used. The one-to-one water-to-solid leach will remain unchanged. The use of the pH paper can be done in the glovebox and will allow mitigation of personnel exposure.

## 3.5 REPORTING

Reporting requirements for data include documentation of activities conducted at PFP, during transport of the sample, as well as in laboratory reports. The following discussions present the documentation required for this SAP. All reports shall be delivered to the Fluor Hanford PFP Residues Project Manager. Reports will be made available to the DOE upon request; transmission of reports to DOE will be from the Fluor Hanford Program Manager only. If reports are provided to the State or federal regulatory agencies, RL will transmit the reports officially.

### **3.5.1 Documentation and Data Package**

#### **3.5.1.1 Field Documentation**

All sampling activities shall be documented in work packages or other controlled documentation packages, maintained by sampling personnel. This documentation must include:

- Identification of the sample source,
- Any observed anomalies, corresponding sample identification numbers, or operational parameters potentially affecting the sample,
- Any conditions observed by the sampling team during the sampling event (e.g., odors, nearby activities, machinery, electrical anomalies),
- Names and titles of personnel involved in the sampling activity and their responsibilities, and
- Problems and procedural changes potentially affecting the validity of the sample.

#### **3.5.1.2 Laboratory Documentation**

Hard-copy laboratory reports will be in two formats, for the purpose of this SAP:

1. Preliminary summary of PCB results to be submitted via FAX to the data quality assessment contractor at 509-946-4595.
2. Full data package capable of undergoing the highest level of data validation.

The required final analytical report (Item 2 above) for analysis of PCBs as Aroclors in the oil from the Turnings in Oil subgroup is defined by the 222-S Laboratory as a Format V report. The contents of the report shall be presented in a manner to allow validation of the data.

The data package shall be issued to PFP Project Management through the document control system. After transmission to and review by PFP Project Management, the raw data shall be accessible to the State and federal regulators until the material has been transported to WIPP.

The data package should be organized into two major parts: (1) a summary report section, and (2) a raw data compilation. Both data package sections will be organized according to the type of analyses or activity that generated the data. The summary report section should be comprised of two subsections: (1) a narrative describing the methods used and any unusual sample or QC results from each analysis or activity, and (2) summary tables of the sample analyses and QC results. Each raw data activity should be organized by analysis type and batch or by the time period when the activity occurred. For most analytical measurements, the batch arrangement should require the least duplication.

### **3.5.2 Electronic Deliverables**

The 222-S Laboratory shall prepare the final data report in electronic format. The electronic format shall be capable of being electronically down loaded and shall be an ASCII, comma-delimited file that is compatible with Excel 1997.

### **3.5.3 Data Validation Report**

The validation reports will be provided based on WHC (1993), Reporting Requirements. The reports will include:

- Introduction,
- Summary of whether project-specific DQO were met,
- Major Deficiencies,
- Minor Deficiencies, and
- References.

### **3.5.4 Data Quality Assessment Reports**

Assessors will provide a letter report to the PFP Residues Project Manager which addresses the following topics:

- Summary of the data,
- Identify data that are missing, incomplete, or are inadequate for decision making,
- Evaluation of the data, and
- Summary of the utility of the data to make the decisions listed in Step 2 of the DQO Process.

## **3.6 DATA REVIEW AND VALIDATION**

### **3.6.1 Data Review**

Data obtained from the pH determination will be peer-reviewed by a person knowledgeable of the requirements identified in the SAP and this QA project plan. Subsequently, copies of the logbook pages will be provided to the PFP Residues Project Manager and the data quality assessment staff.

For the PCB data, the laboratory will perform a peer, or one-over-one, review of all analytical data by a person trained in each particular analytical method being reviewed. HASQARD, Volume 3, Section 8 (DOE-RL 1998), describes this data review. The laboratory also will use its own procedures which conform to HASQARD to provide review of the data before reporting the data to the PFP Residues Project Manager. This review will be performed on all data before submission of the final report to the PFP Residues Project Manager.

After QA review, the Laboratory Project Coordinator will provide a preliminary report of the PCB data to the PFP Residues Project Manager and to the data quality assessment staff. The preliminary report will include:

- Description of the sample material,
- The PCB results by Aroclors,
- Summary of QC data, including method blanks, LCSs, analytical duplicates, and surrogates.

### 3.6.2 Validation of PCB Data for Pourable Oil

Level D validation will be performed according to WHC (1993), modified to include the criteria specified in this SAP. The Level D validation includes:

- Verification of deliverables versus requirements,
- Verification of transcription errors,
- Evaluation and qualification of results on method blanks,
- Evaluation and qualification of results on LCSs, laboratory duplicates,
- Evaluation of initial and continuing calibration, and
- Calculation checks of both sample and QC parameters at a frequency of 20%, or at least one sample and one complete QC sample series, will be recalculated, whichever is greater. A QC sample series is defined as initial and continuing calibration standards, method blanks, spike samples, surrogates, duplicates, and LCSs.

### 3.7 DATA QUALITY ASSESSMENT

Data quality assessment will be performed on the PCB data for pourable oil and the pH determination for the Sludge.

Data quality assessment is performed after data validation. The purpose of data quality assessment is to evaluate whether original project objectives are met, identify data deficiencies that impact data interpretation, and determine whether data are sufficient and of appropriate quality to allow the decisions to be made. The data quality assessment will be performed in a manner consistent with EPA (1996) and the American Society of Testing and Materials (ASTM 1998) and includes the following steps:

1. Review the data with respect to the project DQO. This includes review of the conceptual model and any assumptions that are included in the data collection design. Determine whether the data are consistent with the conceptual model. If the data differ from the model, the decision-makers and technical staff must determine the consequences of using a different model and the impact this has on the decision.
2. Calculate *concentrations*, if required for decision-making.
3. Examine the data for outliers or anomalous values. This includes identification of statistical outliers and anomalous values. Any anomalous values should be validated and closely examined to assess potential reasons for the anomaly. If no reason can be found to exclude the data in question, those data should be included in further analysis. If a reason for exclusion can be found, a detailed but concise explanation for exclusion should be provided.
4. *Evaluate the decision error. Because a judgmental design was performed, decision error cannot be evaluated.*

5. The project records for the sampling of the pourable oil and Sludge, as well as the records supporting the pH determination, will be examined with respect to the project DQO, requirements of the sampling and analysis, and this QA project plan.

#### 4 REFERENCES

- ASTM, 1989, *Test Method D4980-89 Standard Test Methods for Screening of pH in Waste*, ASTM D4980-89, American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- Bailey, W.J., 1958, *Fabrication of Aluminum-Plutonium Alloy Fuel Elements for Lattice Tests in Support of PRTR*, HW-51855, February 3, 1958.
- Bailey, W.J., 1959, *Fabrication of Aluminum - Plutonium Alloy Fuel Elements by Co-extrusion*, HW-63151, December.
- Bailey, W.J., 1961a, *Investigation of Plutonium - Bearing Fuel Elements for the Plutonium Recycle Test Reactor (PRTR)*, HW-SA-2176, June.
- Bailey, W.J., 1961b, *Investigation of Plutonium - Bearing Fuel Elements for the Plutonium Recycle Test Reactor, Part I*, HW-70158 PT I.
- Bloomster, C.H., 1960, *Casting Aluminum-Plutonium Alloys*, HW-63166, March 21, 1960.
- Bloomster, C.H., 1962, *Nuclear Fuel Materials - A Plutonium-Zirconium Alloy - Report of Invention*, HWIR-1524, June 4, 1962.
- Borisch, R.R. et al, 2001, *Plutonium Alloys Report*, HNF-7619, Rev. 1, Fluor Hanford, February.
- Bradley, J.G., 1960, *PRTR Fuel Requirements for Pre-Startup Tests and Initial Operations (Construction End through November 1962)*, HW-63305, January 4, 1960.
- Bradley, J.G., 1961, *Pu-Al Mark I Spike Materials Standard*, HW-71676, November 30, 1961.
- Burnham, J.B., 1959, *Fabrication of Aluminum - 1.8 w/o Plutonium PCTR Loading*, HW-62821, November 11, 1959.
- Chain of Custody for Environmental Media and Waste Samples*, Fluor Hanford Analytical Services Procedures, ASP-200, Rev. 1, June 6, 2000.
- Coffinberry, A.S. (Ed.), 1961, *The Metal Plutonium*, University of Chicago Press.
- Cooper, T.D., 2001, "Stability of Selected Plutonium Alloy Residues", Fluor Global Services, Memorandum to C.S. Sutter 1AC00-PPSL-01-034, July 16, 2001.
- Dienes, N.S., 1985, *Specifications for Plutonium Shipped to the Rocky Flats Plant*, Department of Energy, Albuquerque Operations Office, March 22, 1985.
- EPA, 1997, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, 3<sup>rd</sup> Edition with Updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

- Ewalt, J., 2001, "Dose Rates", email communication, July 12, 2001.
- Freshley, M.D., 1957, *Plutonium-Aluminum Fuel Element Development*, HW-52457, September 18, 1957.
- Freshley, M.D., 1961a, *Plutonium Spike Fuel Elements for the PRTR. Part II – The Mark I-H*, HW-69200 PT II, March.
- Freshley, M.D., 1961b, *The Reactivity of High-Exposure Plutonium*, HW-69169, April 6, 1961.
- Freshley, M.D., 1961c, *Plutonium Spike Fuel Elements for the PRTR. Part I – The Mark I-G*, HW-69200 PT I, March.
- Freshley, M.D., 1964, *The PRTR High Power Density Core Program: Operation of a Substantial Number of Fuel Elements with Molten Cores*, HW-84528, November 24, 1964.
- Freshley, M.D., 1965, *The PRTR High Power Density Core Program Molten Core Operation of Fuel Elements*, BNWC-23, February 12, 1965.
- Hanthorn, H.E., 1963, *PRTR Light Water Injection Capacity*, HW-78159, July 15, 1963.
- Isaacson, R.E., 1963, *Supplemental Specifications for Storage and Processing of PRTR 1.8 Percent Plutonium-Aluminum Alloy Fuel in REDOX Plant*, HW-76605, February 18, 1963.
- Katayama, T.B., 1958, , *Decontamination of Aluminum Plutonium Alloys*, HW-54735, January 28, 1958.
- Kelly, W.S., 1961, *PRTR Fuel Element Examination Facility Operations Manual*, HW-70880, October 9, 1961.
- Kingsley, R. S., 1962, *Recovery of Plutonium from Fabrication Oil*, HW-74350, August 1, 1962.
- Knecht, R.L., 1959, *Fabrication of the PRTR Zircaloy-2 High Pressure Process Tubes*, HW-60358, July 31, 1959.
- Koler, R.K., 1961, *Pneumatic Injection Casting of Aluminum Plutonium Fuel Elements - Report of Invention*, HWIR-1506, March 24, 1961.
- Koler, R.K., 1963, *Irradiation Testing of Injection-Cast, Al-Pu -Ni Alloy Fuel Rods*, HW-SA-2909, February 8, 1963.
- Lyon, W.L., 1957, *Application of the Aluminum Cryolite Reduction Process to the Plutonium Recycle Program*, HW-52461, September.

- Merker, L.G., 1963a, *Hanford Plutonium Fuel Development Laboratory*, HW-SA-2904, February 13, 1963.
- Merker, L.G., 1963b, *Plutonium Fuel Casting Facility – Abstract from Transactions of the American Nuclear Society*, November.
- Merker, L., 2001, Personal Communication with L. Oates, January 8, 2001.
- Merker, L.G., *Plutonium Fuel Casting Facility*, HW-SA-3029.
- Moeller, R.D., 1950, *Alloys of Plutonium with Aluminum*, LA-1000, February.
- NRC/EPA, *Joint NRC/EPA Guidance on Testing Requirements for Mixed Radioactive and Hazardous Waste*, 62 FR 62079, November 20, 1997
- Pankaskie, P.J., 1964, *PRTR Process Tube Monitoring Equipment Description and Operation*, HW-80780, March 1, 1964.
- Sharp, R.E., 1960, *Fabrication of 10 Weight Per Cent Plutonium –Aluminum Monitoring Foils and Pin*, HW-63558, January.
- Smith, R.I., 1962, *Experience with the Plutonium Recycle Test Reactor*, HW-SA-2749, September.
- Staff, 1962, *Startup History of the Plutonium Recycle Test Reactor*, HW-72767, November.
- Tate, R.E., 1961, *The Fabrication of Billets Containing Plutonium for MTR Fuel Elements (from The Metal Plutonium)*.
- “Test Plan for Stability Characterization of Plutonium Alloy Residues”, Letter, February 12, 2001, or most recent update.
- Thomas, I.D., 1960, *Summary of Aluminum –Plutonium Fuels Development at Hanford*, HW-SA-1775, Presented at Conference in April.
- Wauchopé, K.L., 1961, *The Preparation of Plutonium – Aluminum Alloy Fuel Elements for the NRX Reactor (from The Metal Plutonium)*.
- Rector, J. H., and W. B. Weihermiller, 1961, *Tapping Small Holes In Unalloyed Plutonium*, HW-63266.
- Wick, O.J., 1957, *Quarterly Report -January, February, March, 1957 – Plutonium Fuels Development - Plutonium Metallurgy Operation*, HW-51853, September.
- Wick, O.J., 1958, *Quarterly Report -January-March 1958 – Plutonium Fuels Development - Plutonium Metallurgy Operation*, HW-57343, September 4, 1958.

Wick, O.J., (Ed.), 1967, *The Plutonium Handbook*, Gordon and Breach, Science Publishers.

HNF-9138

Rev. 0

**APPENDIX 1. PHOTOS OF GROUP 2B ITEMS EVALUATED AT PFP AS PART OF  
STABILITY ASSESSMENT**

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Rev. 0

**Photo 1. Turnings in Oil (showing dried oil)**



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**Photo 2. Oil Dried on Sides of Can**



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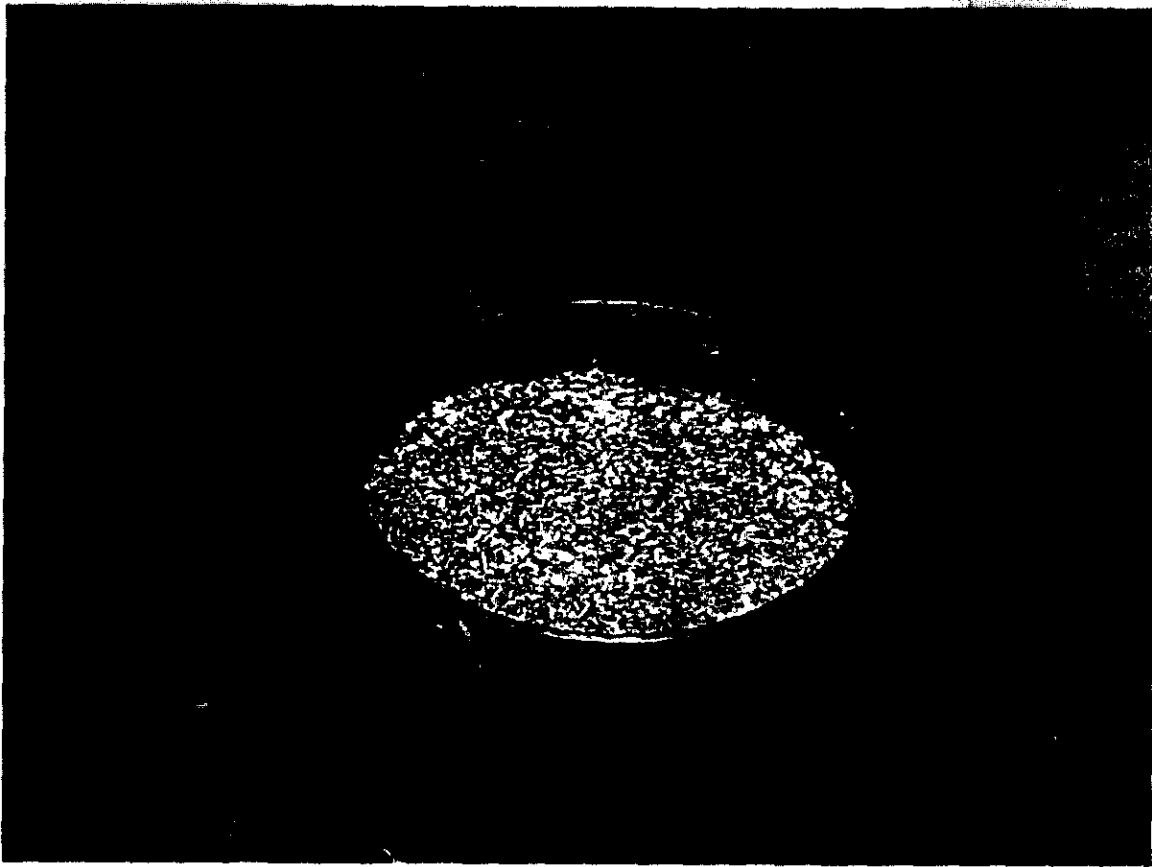
**Photo 3. Skulls in Can**



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**Photo 4. Chips in Can**



## **APPENDIX 2. LOGIC FOR REMOVAL OF CONSTITUENTS OF POTENTIAL CONCERN**

The table attached as Appendix 2 summarizes information about TC organic COPCs that have been eliminated from consideration for the Pu Alloys, Group 2b. The table entries include the waste number (commonly known as "waste code"), waste constituent, selected synonyms for the constituent name, type of organic compound (that is, pesticide/herbicide, volatile, and/or semivolatile) and uses and/or physical characteristics of the waste constituent. The uses are not an exhaustive listing, but include the more common current and former uses of the chemicals to justify why the waste constituent is unlikely to have been associated with the Pu Alloys under consideration in this DQO. The entries are alphabetized by the name of the waste constituent.

In general, pesticides (including insecticides, herbicides, and fungicides) and disinfectants are unlikely to have been associated with the metallurgical and fabrication processes for the Pu Alloys. For some chemicals, the uses and the physical characteristics, such as melting point, boiling point, or explosive nature, eliminated the chemical from consideration. When an identified use is as a degreasing or metal cleaning agent, the final evaluation was to examine whether the available literature associated with the Pu Alloys identified the chemical in association with metallurgical or fabrication processes.

## Logic for Removal of COPCs (5 pages)

Waste Number	Waste Constituent	Selected Synonyms*	Organic Compound			
			Pesticide/ Herbicide	Volatile**	Semivolatiles	Uses and/or Physical Characteristics*
D018	Benzene			X		Solvent for inks, waxes, resins, oils, natural rubber; in manufacture of polymers, detergents, pesticides, pharmaceuticals, dyes, plastics, resins, explosives; as paint thinner, dry-cleaning solvent, gasoline additive, and degreasing agent. Not mentioned in references related to alloys or fabrication.
D020	Chlordane		X			Insecticide; formerly widespread use on agricultural and garden crops, turf, ornamentals, fruits including citrus, nuts, as well as subsurface ground insertion for termite control.
D021	Chlorobenzene	Benzene chloride		X		Dye carrier in textile processing; solvent for paints, bitumen and asphalt coatings for buildings, surface coatings, and surface coating removers; in dry-cleaning, as heat transfer medium; as tar and grease remover in cleaning and degreasing operations. Not mentioned in references related to alloys or fabrication.
D022	Chloroform	Trichloromethane; methane trichloride	X	X		Solvent for fats, oils, rubber, alkaloids, waxes, latex gum, resins; in fire extinguishers; in rubber industry; lubricant additive; as flotation, antifoam, dry-cleaning, cleansing, and flavoring agents; insecticidal fumigant for stored barley, corn, oats, popcorn, rice, rye sorghum and wheat. Not mentioned in references related to alloys or fabrication.
D023	o-Cresol	2-Methyl phenol; 2-hydroxytoluene; 1-hydroxy-2-methylbenzene	X		X	Industrial solvent; metal degreasing agent; in disinfectants and fumigants. Not mentioned in references related to alloys or fabrication.

## Logic for Removal of COPCs (5 pages)

Waste Number	Waste Constituent	Selected Synonyms*	Organic Compound			
			Pesticide/ Herbicide	Volatile**	Semivolatile	Uses and/or Physical Characteristics*
D024	m-Cresol	3-Methyl phenol; 3-hydroxytoluene	X		X	In disinfectants and fumigants; as disinfectant, bactericide, germicide against bacteria in households, sickrooms, hospitals, and veterinary clinics, on surgical instruments, diagnostic equipment, and rubber and plastic items; insecticide and miticide on dogs for treatment of lice and fleas; bactericide for fruit trees and vines; as industrial solvent; in photographic developers and explosives. Not mentioned in references related to alloys or fabrication.
D025	p-Cresol	4-Methyl phenol; 4-hydroxytoluene			X	Industrial solvent; metal cleaning agent; solvent for wire enamels; agent in ore flotation; in synthetic flavor. Not mentioned in references related to alloys or fabrication.
D026	Cresol	Methyl phenol	X		X	In disinfectants and fumigants; in making synthetic resins; as lubricating oil additive; as industrial solvent; wide use in degreasing compounds and paintbrush cleaners; melting points less than 35.5°C, boiling points from 191 to 203°C, and flashpoints less than or equal to 86°C. Not mentioned in references related to alloys or fabrication.
D016	2,4-D (Dichlorophenoxy acetic acid)		X			Herbicide
D027	1,4-Dichlorobenzene	Para-dichlorobenzene	X	X		General insecticide and fumigant, germicide, and space deodorant especially in restrooms; most common domestic use as insecticide against clothing moths.

Logic for Removal of COPCs (5 pages)

Waste Number	Waste Constituent	Selected Synonyms*	Organic Compound			
			Pesticide/Herbicide	Volatile**	Semivolatile	Uses and/or Physical Characteristics*
D028	1,2-Dichloroethane	ethylene dichloride	X	X		Solvent for rubber, asphalt, bitumen, fats, oils, waxes, gums, and resins; in degreaser compounds, rubber cement, acrylic adhesives, soaps and scouring compounds, wetting and penetrating agents, and paint, varnish, and finish removers; for ore flotation; leather and dry cleaning, and pickling and metal cleaning; as a lead scavenger in antiknock gasoline; fumigant for grain, mushroom houses, upholstery, and carpeting. Not mentioned in references related to alloys or fabrication.
D029	1,1-Dichloroethylene	1,1-dichloroethene; 1,1-DCE		X		In adhesives and polymers for food containers and saran plastics; as component of synthetic fibers.
D030	2,4-Dinitrotoluene	1-methyl-2,4-dinitrobenzene			X	Gelatinizing and waterproofing agent in explosives; as an explosives intermediate; in rubber, chemical, and plastics manufacture; solid at 15°C and normal (1 atmosphere) pressure; spontaneously decomposes above 280°C; explodes if confined (decomposition is self-sustaining and does not require air or oxygen); explodes at lower temperatures if under pressure; impact sensitive and may explode upon impact; explosive energy of 2,4-dinitrotoluene is approximately 85% of that of TNT (i.e., trinitrotoluene or dynamite).
D012	Endrin		X			Insecticide
D031	Heptachlor and its epoxides		X			Insecticide against cotton boll weevil, for dipping roots or tops of non-food plants, and for subsurface insertion for termite control.
D032	Hexachlorobenzene	Perchlorobenzene	X		X	Fungicide, especially for seed treatment; as additive in pyrotechnic compositions for the military; in organic synthesis.

## Logic for Removal of COPCs (5 pages)

Waste Number	Waste Constituent	Selected Synonyms*	Organic Compound			
			Pesticide/Herbicide	Volatile**	Semivolatile	Uses and/or Physical Characteristics*
D034	Hexachloroethane	Perchloroethane			X	In metallurgy for refining aluminum alloys, removing impurities from molten metals, and recovering metal from ores or smelting products [although unlikely to persist through fabrication process because it is solid at ambient temperature with melting point of 185°C and boiling point (i.e., triple point) of 186.8°C, that is, it sublimates or becomes a gas, without melting]; as smoke generator in grenades and in pyrotechnics; an ignition suppressant in fire extinguisher fluids. Not mentioned in references related to alloys or fabrication.
D013	Lindane		X			Insecticide
D014	Methoxychlor		X			Insecticide
D035	Methyl ethyl ketone	Butanone; methyl acetone		X		Solvent for vinyl, nitrocellulose, acrylic, lacquer, and varnish coatings; in paint removers, cements, and adhesives; in printing inks and cleaning solutions; as a sterilizer for bacterial spores on surgical instruments, hypodermic needles and syringes, and dental instruments. Not mentioned in references related to alloys or fabrication.
D036	Nitrobenzene				X	In soaps, shoe polishes, and perfumes; as preservative in spray paints; as substitute for almond essence; as an extraction solvent in refining petroleum-based lubricating oils.
D037	Pentachlorophenol		X			Insecticide for termite control; as general herbicide and as a pre-harvest defoliant; in preservation of wood, wood products, starches, dextrans, and glues.
D038	Pyridine				X	Solvent for anhydrous mineral salts; in seafood and smoke flavors and chocolate; melting point of -41.6°C, boiling point of 115.2 to 115.3°C, and flashpoint (closed cup) of 20°C (68°F); autoignites at 482°C; highly explosive when exposed to heat or flame.

## Logic for Removal of COPCs (5 pages)

Waste Number	Waste Constituent	Selected Synonyms*	Organic Compound			
			Pesticide/Herbicide	Volatile**	Semivolatile	Uses and/or Physical Characteristics*
D015	Toxaphene		X			Insecticide
D040	Trichloroethylene	Acetylene trichloride		X		Solvent in printing inks, paint, lacquers, varnishes, adhesives, paint strippers, and some typewriter correction fluids; in metal cleaning and degreasing of furniture and fixtures, fabricated metal products, electric and electronic equipment, transport equipment and miscellaneous manufacturing industries; in vapor cleaning and degreasing. Mentioned in references related to metals fabrication, i.e., cleaning of sheathing tubes and of cans and lids before tinning, which do not relate specifically to the materials in Group 2b.
D041	2,4,5-Trichlorophenol	2,4,5-TCP	X	X		Fungicide and bactericide in cooling towers, paper and pulp mill systems, on swimming pool-related surfaces, hospital rooms, sickroom equipment, bathrooms, food processing plants and equipment, food contact surfaces; antifungal agent in adhesives; preservative.
D042	2,4,6-Trichlorophenol	2, 4, 6-T	X	X		Herbicide; defoliant; fungicide, antimildew treatment for textiles; bactericide; sanitizer; preservative for wood and glue.
D017	2,4,5-TP (Silvex)		X			Herbicide

\*Information adapted from The Merck Index, 12th Edition (1996) or the Hazardous Substances Data Bank of the National Library of Medicine (<http://toxnet.nlm.nih.gov>); all possible uses are not listed.

\*\* Volatile compounds are unlikely to be present at this time in the materials under consideration because they have been stored for such an extended period (i.e., up to 30 years) in unsealed or loosely covered containers.